









**I.—GENERAL; PLANT; MACHINERY.***Settling of precipitates.* Deerr. See XVII.*Automatic separator.* Watson. See XX.**PATENTS.***Incrustations in evaporators and other liquid heaters; Process of preventing gypseous* — Aktieselskapet de Norske Saltverker. E.P. 131,279, 11.7.19. Conv., 14.8.18.

DURING the evaporation of sea-water and other saline solutions a small quantity of fine gypsum crystals is introduced into the liquor to form nuclei for the deposition of more gypsum, whereby, in conjunction with a sufficiently rapid motion of the liquor the gypseous deposits may be removed as mud instead of forming scale. If the liquor contains sulphates, such as magnesium sulphate, the primary addition may take the form of lime.

—B. M. V.

*Scale in boilers and evaporators; Process for removing calcareous* — E. Möller. G.P. 326,019, 24.12.16.

A solution of kieserite and hydrochloric acid is introduced into the boiler whereby the calcium carbonate is converted into calcium sulphate and the resulting increase of volume causes softening and disintegration of the scale.—L. A. C.

*Evaporator.* F. W. Kreutter. U.S.P. 1,358,165, 9.11.20. Appl., 16.1.19.

AN evaporator comprises a furnace with combustion chamber behind, and above these a front and a rear pan section, the front pan section having a rear wall inclined forwards and being connected at its rear end, by means of a pivoted conduit, with the front end of the rear pan section.—W. J. W.

*Fractionating apparatus.* Rosanoff Process Co., Assees. of M. A. Rosanoff and H. F. Perkins. E.P. 137,300, 29.12.19. Conv., 23.12.18.

AN apparatus, specially suitable for carrying out the process described in E.P. 9654 of 1912 (F.P. 443,054; J., 1912, 102), comprises a number of nests of sloping tubes, each nest being maintained at a definite temperature (decreasing progressively in the direction of motion of the vapour) by means of an enclosing box through which cooling or heating fluid may be circulated. The vapour inlet being at the bottom and outlet at the top of the series of units, the condensed liquid from any unit will run back to the next hotter one by gravity.—B. M. V.

*Distilling and heating apparatus.* R. S. Mears. U.S.P. 1,358,091, 9.11.20. Appl., 2.1.19.

A BOILER is provided with a condenser the two ends of which are connected with the steam and water spaces of the boiler respectively. A discharge pipe is connected with the pipe which connects the condenser with the water space of the boiler, and is provided with a check valve opening towards the boiler.—W. F. F.

*Pulverulent materials; Apparatus for treating* — A. P. E. Bourdet. E.P. 153,479, 16.12.19.

THE material to be dried or otherwise treated is allowed to fall through a container across which are three sets of tubes. Two sets are perforated and serve for the supply and withdrawal of the treating fluid respectively, whilst the third set is non-perforated and serves for heating purposes.—B. M. V.

*Surface apparatus for the transmission of heat.* J. W. Mather. E.P. 153,509, 9.2.20.

IN apparatus such as surface condensers the bank

of tubes is made to taper so that a more or less sharp edge is presented to the incoming vapour or other fluid, and the cross-section available for passage of vapour is gradually reduced up to the point of maximum width of the outer casing, whilst the maximum depth of tube-bank comes opposite the middle of the inlet passage, where the velocity is highest.—B. M. V.

*Furnaces; Gas fired* — The Dowson and Mason Gas Plant Co., Ltd., and J. Paton. E.P. 153,511, 14.2.20.

THE goods to be heated are passed, e.g., on a moving hearth, through a long chamber, the middle portion of which is provided with burners coming up through the floor and the end portions of which are used for preheating and cooling the goods. The air for combustion enters flues in the walls of the furnace near the middle of their length, passes to the end and back again and then to a long passage across which jets of gas are blown in line with the burner holes in the hearth. The furnace is suitable for annealing tin plates.—B. M. V.

*Precipitating suspended particles from gas; Apparatus for [electrically]* — A. F. Meston, Assr. to Research Corp. U.S.P. 1,357,886, 2.11.20. Appl., 23.2.18.

THE gas to be treated is subjected to the action of a transverse electric field in a flue comprising a perforated and a non-perforated section, and means are provided for intensifying the field adjacent to the perforated section.—J. S. G. T.

*Gas purification [; Electrical—].* H. F. Smith, Assr. to The Smith Gas Engineering Co. U.S.P. (A, B, C) 1,358,030-2, 9.11.20. Appl., 4.8.17. (c) Renewed 4.8.20.

(A) VAPOURS are removed from gases by first removing the condensation nuclei and then condensing the vapours. (B) Gas is subjected to an electrical discharge and then passed through an electrostatic precipitating field. (C) A gas-purifying apparatus comprises a treatment chamber with inlet and outlet, an electrode between the two, and a second electrode between the first and the outlet. Means are provided for producing an electrical discharge adjacent to the first electrode and also by means of the second electrode to set up an electrostatic field of constant polarity.—C. I.

*Gases; Apparatus for separating dust from* — A. Möllinger. G.P. 321,413, 18.6.19.

THE gas passes through a chamber containing groups of horizontal plates for collecting the dust, so arranged that, without interrupting the process, each group can be inclined temporarily to discharge the dust into a receptacle, whence it is removed as necessary.—L. A. C.

*Agitating device.* C. F. Woodhull. U.S.P. 1,358,045, 9.11.20. Appl., 13.2.20.

AN annular mixing pan rotates and the material therein is stirred by fixed ploughs and discharged by scrapers through a central outlet which is opened when the inner cylindrical wall of the pan is lifted. Means may be provided for simultaneously lifting the wall and lowering the scrapers.—B. M. V.

*Retort.* P. Reecke. U.S.P. 1,358,327, 9.11.20. Appl., 17.10.17.

MATERIAL is fed into the upper end of an inclined retort arranged within and rotating with a concentric outer drum. The material is discharged at the lower end into a cooling receptacle without exposure to the air, and then passes into a water-jacketed receptacle provided with a stirring device.

—W. F. F.

*Filter, cleaner, or washer for air.* J. Rudloff. U.S.P. 1,358,333, 9.11.20. Appl., 19.4.20.

AIR is drawn upwards through liquid contained in a vessel and then passes through a layer of filtering material, supported on a plate above the liquid. The density of the filtering material can be varied by means of a hand screw, acting upon a bowed elliptical spring, which presses a plate upon the filtering material.—W. F. F.

*Drying process and apparatus therefor.* C. Field, Assr. to Chemical Machinery Corp. U.S.P. 1,358,431, 9.11.20. Appl., 8.12.17.

A HORIZONTAL drying chamber suitable for drying plastic or semi-liquid compositions is provided with an end closure carrying a conveyor for the material within the chamber. The conveyor is bodily removed upon removal of the closure, and is automatically placed in or out of connexion with the driving means when inserted into or removed from the drying chamber.—W. F. F.

*Drying tower.* W. L. McLaughlin. U.S.P. 1,359,188, 16.11.20. Appl., 17.1.19. (Cf. U.S.P. 1,336,364 of 1920; J., 1920, 395 A.)

COMMUNUTED material is supplied in a uniform sheet at the top of a tower by means of a spiral conveyor and a casing with a spiral edge, and falls through an upward stream of heated air, being deflected by staggered baffles on its way.—B. M. V.

*Drying chamber.* Internationale Ges. für Trockenanlagen m.b.H. G.P. 324,555, 22.7.15.

THE drying air enters the chamber in front at the bottom and passes out behind at the top. The material to be dried is placed on horizontal perforated partitions within the chamber.—L. A. C.

*Crucible [; Double walled —].* A. E. Bellis, Assr. to Collins and Roessel, Inc. U.S.P. 1,358,816, 16.11.20. Appl., 10.12.19.

A CRUCIBLE has an outer wall resistant to oxidising gases and a removable non-refractory lining resistant to chemical salts.—B. M. V.

*Pulverising mill.* J. W. Fuller. U.S.P. 1,358,837, 16.11.20. Appl., 27.8.17.

GRINDING is effected by freely moving balls running against a fixed ring. The mechanism which drives the balls is provided with scoops or other means to raise the ground material out of the grinding zone and with other scoops to discharge it.—B. M. V.

*Thickening mixtures; Apparatus for continuously —.* A. L. Genter, Assr. to General Engineering Co. U.S.P. 1,359,162, 16.11.20. Appl., 30.4.19.

A TANK with an outlet at the bottom is provided with a number of internal filter elements through any number of which filtrate may be withdrawn, while others may be cleaned simultaneously and while still submerged.—B. M. V.

*Filling material for reaction and absorption apparatus; Tubular —.* H. Frischer. G.P. 324,441, 22.12.18.

THE tubes are split and bent in a suitable shape so that they fit into one another and provide an increased reaction surface.—L. A. C.

*Absorption, scrubbing, and reaction tower.* H. Frischer. G.P. 324,921, 30.9.19.

THE bottom of the tower is divided into compartments, each of which receives the liquid trickling down a different section of the tower, and by measuring the volume of liquid discharged from each section it is possible to ascertain whether the liquid is passing uniformly down the tower.

—L. A. O.

*Sludges; Process for draining — on a filter.* T. Steen. G.P. 324,866, 21.10.16.

THE suction on the filter is regulated automatically according to the permeability of the sludge to air and water and the thickness of the layer on the filter.—E. H. R.

*Furnaces; Mechanical feeding —.* G. Gröndal. U.S.P. 1,360,210, 23.11.20. Appl., 20.4.18.

SEE E.P. 115,639 of 1918; J., 1919, 78 A.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coals; Certain chemical aspects of the South Wales — and coal-field.* S. R. Illingworth. Joint Meeting of Fuel Economy Committee of Brit. Assoc. and S. Wales Inst. of Eng., 26.8.20. [Advance copy.] 25 pp.

THE temperatures at which active decomposition commenced when typical South Wales coals were heated were found to increase with the increase of the carbon-hydrogen ratio in the coals. The behaviour of a coal below 500° C., it is suggested, determines its characteristic properties and economic uses. The different species of coal are differentiated by the amounts of pyridine-soluble constituents they contain, the thermal stability of these substances, their stability relative to one another, and their nature. From the results it is concluded that it should be possible to produce from highly bituminous coals any desired type of semi-bituminous, dry steam, or anthracite coal, by a process of fractional decomposition. It seems probable that Seyler's classification (Proc. S. Wales Inst. Eng., 21, No. 8 and 22, No. 3) based on the hydrogen content is on a true scientific basis, reflecting the chief substances determining the characteristics of a coal.—W. P.

*Bituminous material; Distillation and gasification of —.* Bube, Braunkohle, 1920, 19, 201—206. Chem. Zentr., 1920, 91, IV., 611.

AN apparatus for the distillation of bituminous material designed to provide a graduated heating, to prevent local overheating, and to produce an increased yield of ammoniacal liquor, consists of a shaft furnace in which the material is heated to successively higher temperatures as it passes down over a series of horizontal partitions. A rotating arm attached to a central shaft sweeps over each partition and, after a complete revolution, the material falls on to the partition below. Air is introduced at the lowest partition, where the coke burns with the formation of carbon dioxide; on the partitions above the carbon dioxide is reduced to carbon monoxide; in the next higher zone, in which the gas is cooled by the addition of gas liquor vapour, distillation takes place; while in the uppermost zone the material is dried. Portions of the residue may be removed from any of the lower partitions, leaving only that necessary for combustion.—L. A. C.

*Moist bituminous lignite; Rational utilisation of —.* T. Limberg. Braunkohle, 1920, 19, 237—239. Chem. Zentr., 1920, 91, IV., 611—612.

DISTILLATION of moist lignite in fireclay retorts or in gas producers is disadvantageous for several reasons, and the use of zone producers, such as those described in G.P. 302,322, 303,954, 313,470, and 322,646 (J., 1918, 331 A; 1919, 890 A; 1920, 441 A, 650 A), is recommended. The formation of pasty nuclear layers is thereby prevented, and no decomposition of the tar takes place, as it is diluted and removed from the apparatus at the time of formation.—L. A. C.

*Ammonium sulphate; Direct recovery of* —. A. G. Cbeal. S. Distr. Assoc. Gas Eng., Nov. 23, 1920. Gas J., 1920, 152, 515.

FAILURES in the process for direct recovery of ammonium sulphate in gas works are often due to inadequate circulation of the liquor in the bubbler or the failure, with hot gas, to exclude tarry matter. The latter forms a scum on the surface of the liquor and both causes lead to loss of ammonia. In the discussion, P. Parrish pointed out that another source of loss is the carrying forward of a fine spray of weak acid solution from the bubbler into the oxide of iron purifiers, necessitating the occasional by-passing of the bubbler and the sacrifice of more ammonia to neutralise the iron oxide.—C. I.

*Ammonium sulphate; Manufacture of neutral* —. E. V. Evans. S. Distr. Assoc. Gas Eng., Nov. 23, 1920. Gas J., 1920, 152, 515—517.

THE process operated by the South Metropolitan Gas Co. consists in condensing the ammonia from the fixed ammonia still, and using the diluted solution to spray the crystals at 75° C. in a centrifuge; 8 galls. of solution containing 0.5% NH<sub>3</sub> suffices to neutralise 6 cwt. of acid salt containing 0.4% H<sub>2</sub>SO<sub>4</sub>. This is 40% of the quantity theoretically necessary, as washing proceeds along with neutralisation. Small quantities of ammonia escape during washing, and the evolution of pyridine necessitates the use of a draughting device. The salt containing 1—1.5% of water is dried by air heated to 150° C. in a tower, the descending salt being distributed by hollow perforated pyramids and staggered triangular bars and flaps. Several cycles are needed to bring the salt down to 0.04% of moisture.—C. I.

*Gas; Calorific power and testing of* —. E. V. Evans. Southern Assoc. Gas Eng., Nov. 23, 1920. Gas J., 1920, 152, 511.

A RECORDING calorimeter, known as the "Caloriscopo," is described. It is a modification of Brady's apparatus (J., 1919, 938A). Its principle depends upon the fact that when air is admitted in gradually increasing proportions to a luminous gas flame, or gas added to a non-luminous Bunsen flame, a point is reached when a luminous tip is just visible in the interior of the flame; the proportions of gas and air required to produce this effect afford a measure of the calorific value of the gas. A determination, accurate to within 1 or 2%, can be carried out in less than 30 seconds.—W. P.

*Montan wax from a central German coal*. R. Pschorr and J. K. Pfaff. Ber., 1920, 53, 2147—2162.

CRUDE montan wax was crushed and extracted successively with ether and acetone; the extracts were separately hydrolysed with alcoholic potassium hydroxide, the potassium salts were converted into the calcium salts, and the latter exhaustively extracted with acetone. The mixture of alcohols and unsaponifiable matter thus obtained was acetylated, and the mixed acetates were separated from unsaponifiable matter by utilising their greater solubility in alcohol, and subsequently from one another by fractional crystallisation from ether-alcohol (1:1); tetracosanyl acetate, m.p. 59° C., ceryl acetate, m.p. 65° C., and myricyl acetate, m.p. 70° C. were thus isolated, from which the corresponding alcohols, m.p. 83° C., 79° C., and 88° C. respectively, were obtained. The ethereal extract yields mainly tetracosanol and ceryl alcohol, whilst the latter is obtained in an almost pure condition from the acetone extract. Isolation of montanic acid from the calcium salt (see above) is effected by treatment with benzene and hydrochloric acid, esterification of the crude acid with ethyl alcohol and sulphuric or hydrochloric acid,

and hydrolysis of the purified ester, m.p. 66.5° C., in acid solution. Montanic acid, m.p. 83.5° C., is most readily isolated from the acetone extract. The ethereal extract contains 26% of free acid (reckoned as montanic acid), 41.8% of montanic ester, and 32.2% of substances of unknown composition; the corresponding figures for the acetone extract are 51.3%, 46.5%, and 2.2%, and for the residue, 0%, 63.5%, and 36.5%. The wax contains therefore 17% of montanic acid (as a maximum), 53% of montanic esters, and 30% of substances of unknown composition (as a minimum). (Cf. J.C.S., Jan., 1921.)—H. W.

*Paraffin wax; Preparation of oxidation products of* —. A. Schaarschmidt and M. Thiele. Ber., 1920, 53, 2123—2143.

CHLORINE was passed into melted paraffin wax at 160° C. in such a manner that the current of gas was finely divided by a Witt's stirrer; the gas was almost completely utilised, and the operation, which proceeded without the aid of external heat, was interrupted when the required gain in weight had been attained. Chlorination did not occur quite uniformly and, in addition to isomeric monochloro-derivatives, dichloro-products were also formed before all the paraffin was attacked. The chlorine was removed by heating the chloro-paraffins with alcoholic potassium hydroxide solution. By oxidation with potassium permanganate, an acid, C<sub>17</sub>H<sub>33</sub>CO<sub>2</sub>H, was isolated (as silver salt) from an olefine which had the bromine value 20.4 and was obtained from a chloroparaffin with 10.6% Cl. The less saturated olefines absorbed oxygen far more rapidly and to a much greater extent. Since the oxidisability of these unsaturated hydrocarbons depends greatly on their state of division the following modifications of the procedure were adopted with an olefine obtained from a chloroparaffin with 32% Cl. The olefine was emulsified in an aqueous solution of sodium palmitate and treated with aqueous potassium permanganate; the latter was rapidly reduced, and reaction was practically complete when the weight of permanganate used was about six times that of the olefine. When the reaction was started with small quantities of sodium palmitate and olefine and further additions of the latter made in proportion as the potassium permanganate was reduced, the amount of crude acid formed was about the same, as was also the relative proportion of fatty acid and unsaponifiable matter, but the acid value of the products was greater than that of those prepared by the first method. When a considerable number of double bonds are present in the olefine, acids which are soluble in water are formed in notable amount, whilst much of the olefine is oxidised to carbon dioxide. Ozone is readily absorbed by the olefines at about 40° C. in the absence of solvent, the amount used corresponding approximately with the quantity of hydrogen chloride eliminated during the formation of the olefine. Fission of the ozonide may be effected with water and the non-acidic aldehydic portion subsequently oxidised with ozone; in this manner 105 g. of higher fatty acids and considerable amounts of lower acids soluble in water were obtained from 150 g. of olefine derived from a chloroparaffin with 19.4% Cl.—H. W.

*Paraffin wax; Oxidation of* — to a true wax in ultra-violet light. A. Grün and T. Wirth. Z. angew. Chem., 1920, 33, 291—292.

IN parallel experiments in which paraffin wax was oxidised for 3½ or 5 hrs. in a current of air at 160° C., ultra-violet light (U.S.P., 1,158,205; J., 1915, 1200) had hardly any influence upon the result. In fact the yield of alcohols and insoluble fatty acids produced was somewhat higher when the oxidation was effected in the absence of ultra-violet light.—C. A. M.

*Aliphatic hydrocarbons; Oxidation of — by nitrogen peroxide.* C. Gränacher. *Helv. Chim. Acta*, 1920, 3, 721—737.

PARAFFIN-WAX, m.p. 50°—52° C., is oxidised by a current of air containing 2% of nitrogen peroxide to substances soluble in sodium hydroxide in 10, 7, 6, and 4 days respectively, at 110°, 120°, 135°, and 150° C. The products are not discoloured and consist of a mixture of acetic, butyric, and higher fatty acids, with about 15% of unsaponifiable oil. In order to determine the function and fate of the nitrogen peroxide, experiments with the pure gas were carried out at 140° C. Under these conditions, the gas is rapidly absorbed, more thorough oxidation is realised in 8—10 hrs., and the colourless gas evolved consists chiefly of nitric oxide. It would not be practicable to use nitrogen peroxide merely as an oxygen-carrier, however, for some nitrogen is lost as nitrogen gas, hydrogen cyanide, and, presumably, nitro-compounds, the alkaline solutions of the oxidised wax being red. When pure *n*-undecane is treated with nitrogen peroxide for 10 hrs. at 140° C., 40% of the hydrocarbon escapes oxidation and the products include no acid higher than monoic acid besides the lower fatty acids. It follows that this method will not furnish any definite acid from a particular hydrocarbon. It is considered probable that the higher paraffins in nature only consist to a small extent of the normal members. (*Cf. J.C.S., Jan.*)—J. C. W.

*Fermentation of cellulose.* Fowler and Joshi. See XVIII.

#### PATENTS.

*Fuel; Artificial* —. A. H. Ege. U.S.P. 1,357,627, 2.11.20. Appl., 12.5.19.

AN artificial fuel consists of powdered fuel and coke breeze bonded with a relatively small proportion of a binder composed of Portland cement, oxychloride cement, and salt. Petroleum is incorporated with the mixture, which is then caused to set hydraulically.

*Coal carbonising furnaces.* H. L. Doherty. E.P. 132,489, 10.5.19. Conv., 7.9.18.

COAL is fed into the top of a vertical retort and travels downwards against the upward flow of a burning mixture of gas and air. The rate of descent of the coal and the proportions of gas and air are so adjusted that a highly heated carbonising zone is maintained at the middle of the height of the retort, and this zone only is provided with a refractory lining. The lower end of the retort wall is perforated, and the charge is supported on an annular plate spaced downwards from the bottom of the wall, while a perforated cone is arranged above the central opening. The heating gas is admitted to the chamber surrounding the bottom of the retort so that it enters the fuel over substantially the whole area of the column. Oils and tar are collected in annular channels above the refractory lining, and gas is drawn off at the top of the retort.—W. F. F.

*Discharging material [coke] at the base of shaft [coal carbonising] furnaces; Means for* —. H. L. Doherty. E.P. 152,965, 10.5.19. Conv., 7.9.18. (*Cf. E.P. 132,489, supra.*)

A FIXED annular shelf is supported under an annular opening at the bottom of the shaft. The opening is formed between the outer wall of the shaft and a fixed cone or other central obstruction. Mechanical means, such as a ring of a diameter rather smaller than the fixed annulus and which is given a gyratory motion, are provided to push the material over both the inner and outer edges of the shelf.—B. M. V.

*Vertical retorts and coke-oven chambers; Method of sealing* —. F. J. Collin, A.-G. zur Verwertung von Brennstoffen und Metallen. E.P. 145,782, 2.7.20. Conv., 28.4.19.

A DISH or cupped plate, into the cavity of which the bottom rim of the retort extends, is filled with granular coke kept moist by a small flow of water. The coke employed is that which, previously used for sealing the cover, falls into the dish when the cover is removed.—W. E. F. P.

*Discharging vertical or inclined retorts or chambers; Means employed in* —. R. and J. Dempster, Ltd., H. S. Knight, and F. J. Siddall. E.P. 153,540, 6.7.20.

THE grate beneath the lower end of the retort consists of two pivoted sections of unequal length, one being a single arm and the other forming part of a rotary, multiple-armed device. The sections are controlled externally and can be operated independently or simultaneously.—W. E. F. P.

*Wood gas producer.* L. Arellana. E.P. 138,362, 28.1.20. Conv., 28.1.19.

A VERTICAL producer, in which combustion takes place in a downward direction, has a conical combustion chamber or shaft and a suspended grate, and is provided with means for the admission of air or the escape of gas through the side and between the grate bars. Beneath the grate is a chamber through which the gas produced passes into a second, lower chamber, communicating with the purifying plant.—W. E. F. P.

*Gas-generating apparatus.* H. J. Kotschevar. U.S.P. 1,357,998, 9.11.20.

OIL is discharged, through a jacketed delivery tube, into molten metal contained in a closed, heated retort. The discharge of oil is controlled by a valve actuated thermally, and varies with the temperature of the molten metal.—W. E. F. P.

*Gas cooler.* Façonseisen-Walzwerk L. Mannstaedt und Co., A.-G., and H. Bansen. E.P. 153,453, 16.10.19.

IN a gas cooler having vertical tubes which project into the gas chamber, the tubes depend from a distributor box to which the cooling medium is supplied, the seal between the box and gas chamber consisting of a curtain on the former which dips into a gutter around the jacket of the latter. The lower ends of the tubes are open and dip into a liquid seal by which the cooling medium, if liquid, is carried away. For use with a gaseous cooling medium the liquid seal is provided with a false bottom, from the gas space beneath which short tubes project through the sealing liquid into the cooling tubes; and apparatus for spraying the cooling tubes internally is arranged within the distributor box.—W. E. F. P.

*Oil; Method of extracting — from shale, coal, or other oil-bearing minerals.* H. Edwards. E.P. 153,663, 9.8.19.

THE material is crushed and, after separation of the bulk of the pyrites by a metallurgical ore concentration process, is carbonised at about 500° C. in a muffle, e.g., a muffled Merton furnace. Sulphur is completely removed from the oil thus obtained during the ordinary refining processes.—L. A. C.

*Mineral oils; Process of purifying* —. J. Smith. E.P. 153,844, 14.4.20.

THE oil is heated under a pressure of 100—150 lb. per sq. in. in the presence of an absorbent such as cotton waste saturated with a strong solution of an alkali hydroxide, carbonate, or bicarbonate.

—L. A. C.

*Mineral oils; Acid refining of* —. H. W. Hutton and F. J. Neilson. E.P. 153,857, 20.5.20.

WASHING with alkali of a mineral oil which has received a heavy acid treatment is facilitated by the addition to the oil of sufficient sulphonated fatty oil, e.g., sulphonated castor oil, to dissolve the sulpho acids from the oil, either before, during, or after the addition of the alkali.—L. A. C.

*Fuel oil; Process for preparing a heavy* —. B. Szelinski. G.P. 299,683, 10.11.16.

A HEAVY fuel oil, lighter than water, from which solids do not separate during use, is prepared by mixing with petroleum products the coal-tar fraction (sp. gr. 1.05–1.07) boiling between 280° and 325° C.—E. H. R.

*Gas-producers; Process for increasing the efficiency and the yield of ammonia in* —. H. Koppers, Assr. to The Koppers Co. U.S.P. 1,360,117, 23.11.20. Appl., 9.9.14.

SEE G.P. 279,550 of 1913; J., 1915, 414.

*Hydrocarbons; Process of and apparatus for the alteration of high-boiling-point — to low-boiling-point hydrocarbons.* F. A. Kormann. E.P. 153,654, 6.8.19.

SEE U.S.P. 1,332,849 of 1920; J., 1920, 326 A.

*Feeding pulverised coal to furnaces [by means of air under pressure].* P. E. Van Saun. E.P. 153,746, 8.10.19.

*[Gas] ovens and retorts; Means for closing the doors of vertical* —. F. J. Collin, A.-G. zur Verwertung von Brennstoffen und Metallen. E.P. 150,305, 9.8.20. Conv., 30.8.19.

*Gas purification.* U.S.P. 1,358,030-2. See I.

*Removing sulphur from gases.* E.P. 153,665. See VII.

*Air-heating stove.* U.S.P. 1,357,675. See X.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Carbonaceous materials; Apparatus for distilling —. Process for carbonising carbonaceous materials. Process for distilling carbonaceous material.* G. W. Wallace. U.S.P. (A) 1,358,662, (B) 1,358,663, and (C) 1,358,664, 9.11.20. Appl., 6.8.18, 23.1.19, 16.6.19.

(A) A VERTICAL retort is provided with a central vertical discharge pipe for gases projecting upwards through the bottom nearly to the top and perforated throughout its length within the retort. The discharge pipe is provided with an annular flange near the bottom of the retort to support the charge and can be detached from the gas-exit main below, which is common to a number of retorts. A travelling crane is provided above the retorts by means of which the discharge pipe and flange carrying the residue of the charge can be withdrawn upwards through the retort. (B) In a retort similar to that described in (A) means are provided for automatically relieving pressure produced in the retort by gas evolved from the material outside the coking zone. (C) In a retort similar to that described in (A) the material in the coking zone is maintained in an "uninterrupted condition" by providing a number of spaced annular supports on the central discharge pipe.—W. F. F.

*Incandescence gas mantles.* H. Pacc. E.P. 153,828, 8.3.20.

A FABRIC for the manufacture of incandescence gas

mantles is composed of chrysotile and ramie thread or other material such as artificial silk. The ramie or other material is preferably shrunk as much as possible before weaving with the chrysotile.

—J. S. G. T.

*Intense light; Production of* —. Siemens-Schuckertwerke G.m.b.H. G.P. 325,875, 6.4.18. Conv., 7.4.17.

A MOLTEN metal such as magnesium, aluminium, or zinc, or copper if a green light is desired, is atomised and burnt in an atmosphere containing a high percentage of oxygen.—L. A. C.

*Röntgen tubes.* Siemens und Halske A.-G. G.P. 325,941, 4.1.18.

THE anticathode is formed of tungsten or molybdenum with a mirror of platinum, iridium, or an alloy of the two. These anticathodes are specially suitable for Röntgen tubes depending on pure electron discharges, working without special cooling and giving very hard rays in large quantities.

—E. H. R.

*Wood preservation.* G.P. 325,543. See IX.

*Esters from pyrolygneous acid.* G.P. 325,639. See XX.

## III.—TAR AND TAR PRODUCTS.

### PATENTS.

*Producer-gas tar; Removal of water from crude* —. O. Heise. G.P. 325,157, 7.10.17.

THE tar is filtered hot, and, after it has cooled, is broken up, e.g., by forcing through sieves, and is then passed in a fluid condition over sloping surfaces or rotating drums.—L. A. C.

*Anthraquinone; Process for the purification of* —. Kinzberger & Co. E.P. 143,885, 27.5.20. Conv., 20.9.17.

A SOLUTION of crude anthraquinone in a neutral solvent, such as chlorobenzene or solvent naphtha, is boiled and agitated in the presence of a purifying agent, such as an alkali or alkaline-earth oxide or carbonate, or an aqueous solution of the same, or dilute or concentrated sulphuric acid. When purification is complete, the anthraquinone solution is separated, and the solvent is removed by steam distillation.—L. A. C.

*Anthracene; Process for the purification of crude* —. Kinzberger & Co. E.P. 141,648, 7.6.20. Conv., 30.10.16.

CARBAZOLE is separated from crude anthracene by agitating a boiling solution of the anthracene in solvent naphtha (h.p. 145° C.) with potassium hydroxide until water no longer distils over, further quantities of solvent naphtha being added if necessary. The solution is separated from the sandy precipitate of potassium-carbazole, and deposits pure anthracene on cooling.—L. A. C.

*Pyridine bases; Manufacture of* —. Farb. vorm. Meister, Lucius, und Brüning. E.P. 146,869, 5.7.20. Conv., 22.6.17.

PYRIDINE bases are obtained by heating paraldehyde with aqueous ammonia. It is advantageous to work under stoichiometric conditions and not raise the temperature above 200° C. For example, 132 pts. of paraldehyde when heated with 255 pts. of 5% aqueous ammonia in an autoclave for 4 hrs. at 140° C., 4 hrs. at 160° C., and finally 4 hrs. at 180° C., gives a mixture of pyridine bases consisting for the most part of 2-methyl-5-ethylpyridine, the yield amounting to about 68% of the theoretical.

—G. F. M.

*Nitro compounds, nitroso and azo compounds, or the like; Reduction of substituted —.* T. S. Moore. U.S.P. 1,358,324, 9.11.20. Appl., 26.7.20.

AROMATIC nitro compounds containing at least one substituent other than a nitro group are rapidly reduced by means of borings of cast iron and an aqueous solution of a chloride.

Fuel oil. G.P. 299,683. See IIA.

#### IV.—COLOURING MATTERS AND DYES.

*Adsorption compounds [of dyestuffs].* III. R. Haller. Kolloid Zeits., 1920, 27, 30—34. (Cf. J., 1918, 411 A; 1919, 281 A.)

THE substrate in the complex Cotton Red 4 BX-lead hydroxide may be changed into lead sulphate, or lead sulphide, without setting free the dyestuff, but conversion into lead acetate, sodium plumbite, lead chloride, or lead chromate at once sets the dyestuff free. From these results two general rules may be deduced. When the substrate is converted into a soluble compound the adsorption complex is destroyed. When the substrate is converted into an insoluble compound which is capable of adsorbing the dyestuff, a new complex is formed without apparent dissociation of the primary complex, but if the insoluble compound has no adsorptive effect on the dyestuff the complex is destroyed and the dyestuff set free.—J. F. S.

Cyanines. Lumière and others. See XXI.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Cellulose; Constitution of —.* K. Hess. Helv. Chim. Acta, 1920, 3, 866—869. (Cf. J., 1920, 512 A.)

A REPLY to criticisms by Karrer (cf. J., 1920, 795 A). (Cf. J.C.S., Jan.)—J. C. W.

*Fermentation of cellulose.* Fowler and Joshi. See XVIII.

#### PATENTS.

*Fibre, and process for obtaining the same [from seaweed].* Y. Hashimoto. E.P. 153,804, 13.1.20.

FIBRES suitable for paper making and as a cotton substitute are obtained from "sugamo" (*Phyllospadix scouleri*, Hook). The exterior of this seaweed is a gum-like material which cannot be separated by mechanical means, but is removed by boiling the raw or dried seaweed with milk of lime (10% CaO) for  $\frac{1}{2}$  to 1 hr. (aerial oxidation must be avoided), washing with water, boiling with a solution of sodium bisulphite of 1° B. (sp. gr. 1.007) for 20 to 50 mins., washing, and rubbing away the softened crust.—A. J. H.

*[Vegetable] fibres; Method of treating —.* H. R. Moody and L. H. Friedburg. U.S.P. 1,357,580, 2.11.20. Appl., 13.1.17.

THE fibres of banana and other tropical plants are rendered suitable for the production of paper pulp or other purposes by immersion in sea water which is being electrolysed, whereby they are disintegrated and bleached.—A. J. H.

*Wool or analogous materials; Process for removing fat from — by the aid of fat solvents.* F. Koch, Assr. to The Chemical Foundation, Inc. U.S.P. 1,358,163, 9.11.20. Appl., 3.2.14.

FAT or oil is extracted from wool or similar fibrous material by treating the latter with a chlorinated hydrocarbon of the fatty series, in absence of soap, soda, or other emulsifying agent.—W. J. W.

*Fabric; Fireproof — and process of producing the same.* C. L. Saunders, G. C. Stanley, and C. W. Bennett. U.S.P. 1,358,250, 9.11.20. Appl., 13.10.19.

FABRICS are rendered fireproof by coating them with a composition containing ammonium sulphate and a hydrated oxide of tin. Cotton fibres are more easily impregnated with sodium stannate if they are first boiled in water, treated with a solution of commercial hydrofluoric acid (2 pts. of acid to 100 pts. of water), washed, and partially dried.

—A. J. H.

*[Wood pulp] digesters.* Aktiebolaget Vaporackumulator. E.P. 141,708, 12.4.20. Conv., 12.4.19.

THE pressure within a digester sometimes exceeds that of the steam supply, and acid is then forced from the digester into the connecting pipes. This disadvantage is overcome by the insertion of a pressure lock between the acid-proof valve nearest to the digester and the non-return valve nearest to the steam supply. The pressure lock consists of a piece of acid-proof tubing arranged vertically and is connected by means of a pipe, having an open valve, to a source of pressure of steam, water or air greater than the maximum attained in the digester. When the pressure within the digester temporarily rises above that of the steam supply, acid liquor flows through into the pressure lock and closes the non-return valve. The acid is thus prevented from entering the steam supply pipes, and rises in the pipe connected with the pressure lock until checked by the greater pressure therein. When the pressure within the digester becomes normal, the acid liquor is forced out of the pressure lock back to the digester, thus allowing the non-return valve to re-open.

—A. J. H.

*Wood; Treatment of — and recovery of organic products therefrom.* H. F. Weiss, Assr. to C. F. Burgess Laboratories. U.S.P. 1,358,129, 9.11.20. Appl., 14.5.18.

WOON of the western larch is comminuted and leached to extract the galactan and other soluble constituents, and the residue is subjected to chemical treatment for the preparation of pulp. The soluble matters are thus obtained uncontaminated with chemicals, and their removal reduces the consumption of chemicals in the subsequent treatment of the wood.—J. H. J.

*Artificial threads, films and other products from viscose; Process for the manufacture of —.* R. Muller. E.P. 153,444, 9.10.19.

By making the processes in the manufacture and use of viscose, cyclic, economy in the consumption of raw materials is possible. The caustic soda is prepared electrolytically from sodium chloride, and the gaseous by-products, hydrogen and chlorine, are suitably combined to form hydrochloric acid, which is used for precipitating the solution of viscose, whereby sodium chloride is formed and is recovered and used for the production of caustic soda.—A. J. H.

*[Viscose;] Method and apparatus for the production of masses or solutions [of —] free from air or other gases.* B. Borzykowski. U.S.P. 1,357,946—7, 9.11.20. Appl., 13.8.17 and 6.4.20.

A FLUID mass of viscose or like material, suitable for the production of artificial threads, films, and the like, is "filmed" on a surface within a vessel exhausted of air. The mass is preferably incorporated with excess of an indifferent highly volatile solvent before treatment. The apparatus consists of a receptacle for containing the fluid mass, two chambers within which the mass is "filmed" under vacuum, and a filter between each chamber and the receptacle. A spinning system is connected with the two chambers and is exhausted of air.

—A. J. H.

*Cellulose acetate; Composition of — with the fatty acids of coconut oil.* G. W. Miles, Assr. to American Cellulose and Chemical Manufacturing Co. U.S.P. 1,357,335, 2.11.20. Appl., 30.6.19.

CELLULOSE acetate is mixed with the total fatty acids extracted from coconut oil.—L. A. C.

*Cellulose-ether composition and method of making the same.* H. T. Clarke, Assr. to Eastman Kodak Co. U.S.P. 1,357,614, 2.11.20. Appl., 17.3.19.

A TRANSPARENT composition contains a cellulose ether and a sulphone.—L.A.C.

[*Cellulose-ester films.*] *Purifying and washing process [for —].* S. E. Sheppard, Assr. to Eastman Kodak Co. U.S.P. 1,357,733, 2.11.20. Appl., 5.6.18.

ACIDIC substances are removed from cellulose-ester bodies, having a large surface in proportion to their mass, by washing them with a strong solution of a salt.—A. J. H.

*Nitrocellulose composition.* J. M. Kessler, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,357,876, 2.11.20. Appl., 6.12.19.

NITROCELLULOSE is incorporated with an alkyl ester of an acyloxy derivative of a higher fatty acid.

*Paper-making stock; Process and apparatus for preparing —.* C. H. Allen and E. J. Trimby, Assrs. to Great Northern Paper Co. U.S.P. 1,357,760, 2.11.20. Appl., 21.8.19.

FLUID mixtures or solutions of the several ingredients of the stock are continuously poured into a common stream in the desired proportions.—A. J. H.

*Separating water or other liquids from solutions [cellulose waste lyes]; Method of —.* E. Oman. U.S.P. 1,359,911, 23.11.20. Appl., 18.9.17.

SEE G.P. 316,592 of 1917; J., 1920, 483 A.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### PATENTS.

*Skein-dyeing machine.* A. T. Scaramuzzi, Assr. to T. J. Corrigan. U.S.P. 1,357,522, 2.11.20. Appl., 4.9.19.

A DYEING machine contains a series of elongated horizontal skein carriers on a horizontal support. A carriage moves from one end of the support to the other, and is so constructed that each skein carrier is in turn displaced from the front to the rear of the advancing carriage.—A. J. H.

*Dyeing machine.* H. M. Dudley. U.S.P. 1,359,043, 16.11.20. Appl., 9.12.19.

THE dyeing machine consists of a receptacle divided into three parts. The middle division forms the dyeing chamber, which is closed at the top and bottom by perforated plates with covers, and contains several removable rods. The upper and lower divisions serve as liquid containers, and each contains a perforated pipe. Means are provided for circulating a liquid in either direction through the perforated pipes and the dyeing chamber.—A. J. H.

*Cotton and cotton manufactures; Process of treating [fireproofing] —.* S. Kashitani. U.S.P. 1,360,224, 23.11.20. Appl., 10.12.18.

SEE E.P. 144,083 of 1919; J., 1920, 542 A.

*Cleaning agent.* E.P. 146,222. See XII.

*Cleaning process.* G.P. 325,796. See XII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Townsend [electrolytic] cell; Chemical efficiency and concentration of caustic soda and salt from —.* A. H. Hooker. Chem. and Met. Eng., 1920, 23, 961—968.

A DESCRIPTION of the plant at Niagara Falls, producing 65 tons of caustic soda and 60 tons of chlorine daily, is given. Types of cell in use are of 2500, 5000, and (most recent) 4000 amp. capacities. A high current density of 100 amp. per sq. ft. of cathode, at a little over 4 volts per cell, is used, with an average current efficiency of 96—97%. The caustic soda concentration is 125 g. per l. Curves showing various data in connection with the cell are given, including specific gravities of brine and caustic soda solutions, solubilities of chlorine in water and brine, chemical efficiencies, and evaporator data.—J. R. P.

*Chlorate; Influence of chloride on the solubility of — and its dependence on temperature.* J. Billiter. Monatsh. Chem., 1920, 41, 287—295.

THE solubility of sodium chlorate in unit volume of either water or sodium chloride solution increases linearly with the temperature, and the diminution produced in the solubility of sodium chlorate by addition of sodium chloride increases as the temperature rises, the extent of the increase being specially great for high concentrations of the chloride. (Cf. J.C.S., Jan., 1921.)—T. H. P.

*Salts having a common ion; Separation of two —.* A. T. Schloesing. Comptes rend., 1920, 171, 977—981.

USING the pure salts, curves are first plotted showing the composition of solutions saturated with respect to the two salts at different temperatures. The solution from which it is required to separate one salt is analysed, and from the data obtained and the curves previously plotted it is possible to determine the temperature at which the solution should be evaporated, so that only one salt will separate out.—W. G.

*Potassium iodide; Titration of — with mercuric chloride.* I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 836—842.

THE corrections which must be made in the titration are shown to be easily calculated by applying the laws of mass action to the balanced reaction,  $\text{HgI}_2 + 2\text{KI} \rightleftharpoons \text{K}_2\text{HgI}_4$ , the complex-constant for this dissociation, and the solubility of mercuric iodide being known. The correction as determined from theory is found to agree exactly with the figures obtained by the use of known solutions. Temperature has very little effect on the end point, and foreign substances interfere only in so far as they have a tendency to form complex mercuric salts. (Cf. J.C.S., Jan., 1921.)—S. I. L.

*Mercuric chloride; Analysis of —.* I. M. Kolthoff and J. Keijzer. Pharm. Weekblad, 1920, 57, 913—919.

REMP's method of reduction by means of formalin in alkaline solution, and estimation of the separated mercury with iodine in acid solution, gives variable results, partly because reduction is incomplete, partly because formalin itself, after heating with alkalis, absorbs iodine in acid solution. Hydrogen peroxide is a more suitable reducing agent, but the method is in any case laborious. Rapid and accurate results may be obtained by adding to the mercuric chloride solution a slight excess of hydrocyanic acid and titrating the solution with caustic soda, using dimethyl-yellow as indicator. Care must be taken that both solutions are neutral to the indicator before mixing, and that



the hydrocyanic acid solution is free from cyanic acid. (Cf. J.C.S., Jan., 1921.)—S. I. L.

**Dolomite; Experimental separation of lime in —.**  
R. T. Stull. U.S. Bureau of Mines. J. Franklin Inst., 1920, 190, 739.

THE presence of lime in dolomite interferes with its use for the manufacture of fire-resistant brick. Treatment of a dolomite containing, after calcining, MgO 42%, CaO 58%, with running water for one day increases the magnesia content to 65% and allows a 50–60% recovery of lime. Boiling the calcined dolomite in water and separating the magnesium hydroxide by flotation gives 65–75% magnesia content, with 25% lime recovery. Addition to the calcined dolomite of sulphuric acid sufficient for all the lime, gives a 75% magnesia content with 50% lime recovery.—B. V. S.

**Ozone; Action of — on alkali metals, ammonia, and substitution products of ammonia.** W. Strecker and H. Thienemann. Ber., 1920, 53, 2096–2113.

WHEN ozone was passed into solutions of the alkali and alkaline-earth metals in liquid ammonia, orange to brown precipitates were obtained, which were very unstable, being rapidly decomposed by water or dilute acids with evolution of oxygen. On this account, and because the precipitates were contaminated with the oxidation products of ammonia, their composition could not be determined. Ozone oxidises liquid ammonia completely to ammonium nitrate (about 98%) and nitrite (about 2%), according to the equations  $2\text{NH}_3 + 4\text{O}_3 = \text{NH}_4\text{NO}_3 + 4\text{O}_2 + \text{H}_2\text{O}$ ;  $2\text{NH}_3 + 3\text{O}_3 = \text{NH}_4\text{NO}_2 + 3\text{O}_2 + \text{H}_2\text{O}$ . The action is retarded but not stopped by drying the ozone as completely as possible. Hydroxylamine hydrate is oxidised by ozone to hydroxylamino nitrate, whilst hydrazine hydrate gives mainly nitrogen and water, with traces of nitrate and ammonia. Methylamine is oxidised to formaldehyde, ammonia, nitrate, and nitrite, and dimethylamine gives in addition formic and acetic acids. Trimethylamine is oxidised explosively even at very low temperatures. By passing ozone into a 5–10% solution of trimethylamine in chloroform, trimethylamine oxide,  $(\text{CH}_3)_3\text{N}_2\text{O}$ , was obtained in the form of its hydrochloride. (Cf. J.C.S., Jan., 1921.)—E. H. R.

**Chlorine and ammonia; Reaction between —. II.**  
W. A. Noyes and A. B. Haw. J. Amer. Chem. Soc., 1920, 42, 2167–2173. (Cf. Noyes and Lyon, J., 1901, 943.)

ANHYDROUS ammonia and anhydrous chlorine react to form nitrogen trichloride and ammonium chloride:  $4\text{NH}_3 + 3\text{Cl}_2 = 3\text{NH}_4\text{Cl} + \text{NCl}_3$ . A considerable proportion of the trichloride always decomposes into chlorine and nitrogen, either directly or by interaction between the trichloride and ammonia. The ratio of the number of mols. of chlorine to the number of mols. of ammonia may be varied between wide limits without affecting appreciably the ratio (4:3) between the number of mols. of ammonia and the number of mols. of ammonium chloride. The chlorine reacts primarily with the ammonia, but only to a trifling extent, or not at all, with the solid ammonium chloride formed. The reaction was carried out with the dry substances without a solvent, and also in presence of carbon tetrachloride or pentane.—J. R. P.

**Chlorine and ammonia; Reaction between —. III. Probable formation of trichloro-ammonium chloride.** W. A. Noyes. J. Amer. Chem. Soc., 1920, 42, 2173–2179.

Dry hydrogen chloride converts nitrogen trichloride quantitatively into ammonium chloride. The reaction may occur either with a solution of the trichloride in carbon tetrachloride or without any solvent. As the decomposition cannot be a hydro-

lysis in the absence of water, it is probably not a hydrolysis in the presence of water. The action probably consists primarily in the formation of trichloro-ammonium chloride,  $\text{NCl}_3\cdot\text{HCl}$ , followed by the loss of an atom of positive chlorine with an atom of negative chlorine. The formation of nitrogen trichloride by the action of chlorine on a solution of an ammonium salt appears to be the reverse of the above reaction. Ammonium sulphate is much more suitable than ammonium chloride for the preparation of nitrogen trichloride. Chloro-amine and dichloro-amine are formed as well as nitrogen trichloride by the action of chlorine on ammonium salts. Treatment of an ammonium salt with hypochlorous acid gives a trichloride pure and free from chloro-amines. Nitrogen trichloride may be separated from a solution in which it is prepared by passing a current of air through the solution, drying the air and vapour of trichloride by passing through concentrated sulphuric acid, and condensing the trichloride in a U-tube surrounded by a freezing mixture.—J. R. P.

**Arsenic; Pure metallic —.** C. H. Jones. Chem. and Met. Eng., 1920, 23, 957–960.

ARSENIC of 99.5–99.7% purity is prepared by heating a mixture of 1 pt. of wood charcoal with 4 pts. of arsenious oxide in gas-fired furnaces holding 200 or 400 lb. of mixture. The furnaces are built up of steel pipe surrounded by firebrick, supported on pipe framework. The cover of the charging end is clamped against a clay gasket with a cooling pipe embedded, which condenses any leak of arsenic and forms a joint. The operation is carried out at a temperature of 650°–700° C., and takes 10 hrs. for the large furnace and 7 hrs. for the small. The chief impurity in the product is arsenious oxide. The arsenic is condensed in a water-cooled condenser at the rear of the furnace. The product has sp. gr. 5.73, is brittle, and may be powdered without difficulty. Among the uses mentioned are as a flux, for addition to copper, bronze, Muntz metal, arsenical lead, and speculum metal.—J. R. P.

**Ammonium sulphate recovery.** Cheal. See IIa.

**Neutral ammonium sulphate.** Evans. See IIa.

**Alkali ferrites and ferrates.** Grube and Gmelin. See XI.

**Transformation of ammonia into urea.** Matignon and Fréjacques. See XX.

#### PATENTS.

**Aluminium oxide; Producing — from aluminium chloride.** S. E. Sieurin. E.P. 153,500, 19.1.20.

Raw material containing aluminium is dissolved in hydrochloric acid and the solution saturated with hydrochloric acid gas, the aluminium chloride which separates being subsequently calcined. To obtain a strong gas suitable for saturating the solution only that evolved at the end of the calcination is taken (a furnace with several beds being used), or the surplus water is separated from the gas by condensation.—C. I.

**Nitrate of soda; Recovery of — from solutions.** I. B. Hobbsawn. In part from J. L. Grigioni. E.P. 153,649, 6.8.19.

A MORE complete separation than hitherto of sodium nitrate from the chloride and sulphate accompanying it is effected as follows. The solution is boiled at 90°–95° C. under an absolute pressure of 11 lb. per sq. in., whereby much of the sulphate and chloride is precipitated. This stage is carried on as a continuous process. The settled liquor is drawn off and further concentrated at atmospheric pressure until the dissolved chloride is equivalent to 75–80 g. NaCl per l. The liquor is then cooled under slightly reduced pressure and crystallised in a rotary cooler.—C. I.

**Potassium and sodium nitrates; Process of separating** — R. P. Calvert, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,356,806, 26.10.20. Appl., 1.8.17.

SOPRUM nitrate mother liquors containing potassium nitrate in quantity equal to at least 20% of the mixed nitrates are subjected to a temperature of 5° C., and the potassium nitrate which separates from the solution is recrystallised.—W. J. W.

**Nitrates and chlorides; Process of leaching and obtaining** — W. E. Giesecke, Assr. to Ray Bros.' Corp. U.S.P. 1,357,973, 9.11.20. Appl., 27.3.17.

THE raw material is mixed with a lixiviating agent, and its disintegration is assisted by blowing a fluid through it under pressure. The finest particles are thus forced above the heavier solids, and the settled material serves as a filter through which the solution is drawn off. Fresh lixiviating agent is then forced through the material from below together with air, and the solution is again filtered and removed.—W. J. W.

**Potassium chloride; Preparation of — from crude, low-grade potassium salts.** Salzwirk Heilbronn, G. Kassel, and T. Lichtenberger. G.P. 325,398, 8.10.18. Addn. to 289,746 (J., 1920, 405 A).

THE melt containing potassium chloride (*loc. cit.*) is heated until the latter is volatilised; it is purified by fractional condensation. If the potassium salts do not already contain chloride, salt or hydrochloric acid is added before heating.—E. H. R.

**Gases; Removal of sulphur [hydrogen sulphide] from** — N. E. Rambush. E.P. 153,665, 11.8.19.

THE gas is washed with a suspension of ferric hydroxide in ferrous sulphate solution. The suspension circulates between the washer and a tower down which it flows against a current of air; or it may be simply run over a system of wooden lattice boards like a water cooler. A portion of the revived suspension is drawn off and the sulphur separated by fractional subsidence or centrifuging.—C. I.

**Arsenical salts; Method of making** — W. Thum, J. J. Mulligan, and H. M. Schleicher, Assrs. to United States Smelting, Refining, and Mining Co. U.S.P. 1,356,569, 26.10.20. Appl., 28.6.19.

INSOLUBLE metallic sulphates are treated with soluble arsenical salts to produce arsenical salts of the metals.—W. J. W.

**Magnesium sulphate; Method of making** — W. R. Collings and J. A. Gann, Assrs. to The Dow Chemical Co. U.S.P. 1,356,907, 26.10.20. Appl., 10.2.19.

MAGNESIUM hydroxide is treated with sulphur dioxide, and the bisulphite formed is oxidised to sulphate.—W. J. W.

**Magnesium carbonate or Magnesia usta; Manufacture of** — W. Esch. G.P. 325,141, 13.10.14.

CALCINED magnesia made to a paste with the requisite amount of water is converted into magnesium carbonate by the action of 1 mol. of carbon dioxide and 1½ mols. of ammonia (or, in the case of *Magnesia alba* which is dense or contains chlorides, of ½ mol. of carbon dioxide and ¾ mol. of ammonia), and is either separated or worked up to light *Magnesia alba* or *Magnesia usta*.—L. A. C.

**Alkali-metal cyanide; Apparatus for manufacture of** — F. J. Metzger, Assr. to Air Reduction Co. U.S.P. 1,358,014, 9.11.20. Appl., 5.12.18.

AN inclined tubular retort has its middle portion

enclosed in a furnace. The charge of an alkali and a carbonaceous material is fed in at the top and nitrogen passed in at the bottom, and by rotating the retort on its axis the lower portion, projecting beyond the furnace, serves as a cooling and nodulising zone for the reaction product.—C. I.

**Alkali cyanide; Apparatus for manufacturing** — F. J. Metzger, Assr. to Air Reduction Co. U.S.P. 1,358,383, 9.11.20. Appl., 25.7.17.

A RETORT of readily oxidisable metal is surrounded by a chamber of material resistant to oxidation, the intervening space being filled with inert gas. Heat is applied to the outer chamber.—C. I.

**Metal [vessels] heated to a high temperature [e.g. in production of cyanides]; Method for protecting — against oxidation.** H. B. Kipper. U.S.P. 1,358,161, 9.11.20. Appl., 19.5.19.

METAL vessels heated to a high temperature, as in the production of nitrogen compounds by heating sodium salts with carbon and iron in presence of nitrogen, may be protected against oxidation by burning carbonaceous fuel to carbon monoxide with a minimum of carbon dioxide, and by avoiding excess of oxygen.—W. J. W.

**Cyanide compound and process of producing the same.** W. S. Landis, Assr. to American Cyanamid Co. U.S.P. 1,359,257, 16.11.20. Appl., 16.1.20.

A MIXTURE of crude calcium cyanamide, an alkaline-earth carbide, and a flux containing less sodium chloride than the molecular equivalent of the cyanide to be formed is heated and then immediately cooled below 400° C. The compound obtained contains a large percentage of calcium cyanide mixed with calcium oxide and fluxing materials.—C. I.

**Sulphur dioxide; Process of reducing** — T. Shiomi, Assr. to Furukawa Gomei Kaisha. U.S.P. 1,359,114, 16.11.20. Appl., 15.1.19.

SULPHUR dioxide mixed with steam and a reducing gas is passed through a heated chamber filled with a catalyst.—C. I.

**Phosphoric acid and compounds of the same; Process of and apparatus for producing** — F. S. Washburn, Assr. to American Cyanamid Co. U.S.P. 1,359,211, 16.11.20. Appl., 21.4.19.

A MIXTURE of phosphate rock and silicious material is preheated in a rotary furnace and then fused in two stages in a hearth furnace. The gaseous products of the last stage are used in preheating fresh charges.—C. I.

**Hydrogen peroxide solutions; Stabilising** — M. Sarason. G.P. 325,861, 3.7.18.

THE addition of 0.2% of a hypophosphite—e.g., sodium hypophosphite—to hydrogen peroxide or solutions of the same prepared from sodium peroxide or perborate prevents decomposition even if the solution is alkaline and maintained at 70° C.—L. A. C.

**Hydrogen sulphide; Separation and recovery of sulphur from solutions, gases, and vapours containing** — Gewerkschaft des Steinkohlen-Bergwerks "Lothringen," and G. Wiegand. G.P. 326,159, 13.12.18.

VAPOURS containing hydrogen sulphide are led, mixed with oxygen, over a solid contact mass sprinkled with a solution of ammonia, alkali, or alkaline-earth, the quantity of oxygen being regulated so that alkali polysulphides are formed, but no free sulphur. As catalysts, oxides and hydroxides of iron, manganese, aluminium, and related metals, or mixtures of these, may be used. The

polysulphides can be decomposed into sulphur and hydrogen sulphide and the latter again put through the process.—E. H. R.

*Alkali amides; Process for the manufacture of —.* O. Liebknecht, Assr. to The Roessler and Hasslacher Chemical Co. U.S.P. 1,359,080, 16.11.20. Appl., 29.1.15.

SEE E.P. 1613 of 1915; J., 1915, 1143.

*Magnesium chloride or anhydrous double chlorides of magnesium; Production of anhydrous —.* E. A. Ashcroft. U.S.P. 1,359,652, 23.11.20. Appl., 12.8.19. Renewed 9.10.20.

SEE E.P. 152,401 of 1919; J., 1920, 783 A.

### VIII.—GLASS; CERAMICS.

*Optical glasses; Relations between chemical composition and refractivity in —.* F. E. Wright. J. Amer. Ceram. Soc., 1920, 3, 783—832.

WHEN the mean optical dispersions and refractive indices of a series of ordinary crown and flint glasses are plotted as ordinates and abscissae, the resulting graph is practically a straight line. When the refractive index and the ratio of the length of the red to that of the blue end of the spectrum are similarly plotted a smooth curve is formed. In fluor-crown glasses the length of the red end of the spectrum exceeds that of the blue end relatively more than in any other glass types; the borosilicate crowns follow next, then the ordinary crowns, barium crowns, and barium flints, and lastly the flints in which the relative dispersion of the blue exceeds that of the red end of the spectrum. From such a graph in combination with a table of analyses the chemical composition of glasses relative to their refringence and relative dispersions can be found. On plotting against one another the partial dispersions for different parts of the spectrum of a series of silicate glasses the resulting graph is a straight line, showing that each partial dispersion of a glass bears a linear relation to any other, so that if one partial dispersion is given, the type of optical glass is fixed, whilst if any two refractive indices of an optical glass are given the dispersion curve can be written down directly, and if two optical glasses of very different indices have the same dispersion for one part of the spectrum their dispersion curves are identical throughout the visible spectrum. These linear relations only hold for that portion of the dispersion curve remote from an absorption band. When the refractive indices are plotted against the squares of the frequency ( $1/\lambda^2$ ) another straight line graph is obtained. With the rise in refractive index the dispersion also rises, the latter rising very rapidly in flint glasses. The chemical compositions of 117 potash-flint, soda-flint, barium-flint, borosilicate flint, barium crown, and borosilicate crown glasses from various sources, when arranged on a triaxial diagram, were found to lie on a straight line uniting the points representing the formulae  $\text{PbO}, \text{SiO}_2$  and  $\text{K}_2\text{O}, 6\text{SiO}_2$ , or the corresponding soda glass  $\text{Na}_2\text{O}, 4\text{SiO}_2$ . The whole of these glasses are analogous to two-component mixtures, and, as such, any one of their physical constants such as refractive index or density varies continuously with a change in composition. In preparing a batch to reproduce glass of given optical properties due allowance must be made for loss by selective volatilisation, suitable allowances being 0.5–5%  $\text{PbO}$ , 1–5%  $\text{B}_2\text{O}_3$ , and 0–5% of alkalis. By means of the foregoing data charts may be prepared from which the batch composition for a glass of any required density, refractive index, and dispersion can be read off directly. Examples of various batches prepared in this manner are given.—A. B. S.

*Porcelain moulded under different conditions; Rate of vitrification of —.* R. F. Sherwood. J. Amer. Ceram. Soc., 1920, 3, 837—841.

TEST-PIECES of a typical porcelain body were made by hand-pressing of the plastic mixture in a plaster mould, by casting in a similar mould, and by dry pressing in a steel mould in an Olsen testing machine using pressures of 2000, 4000, and 6000 lb. per sq. in. respectively. The densest product was that formed by dry pressing at 6000 lb. per sq. in., and the least dense material was formed by hand-pressing of the plastic mixture. After firing at  $1120^\circ\text{C}$ . the initial porosity was lowest in the cast pieces, and greatest in the pieces dry-pressed at 2000 lb. per sq. in. The rate of vitrification was greatest in the plastic-moulded test-piece, this being followed by the cast material. There appeared to be a distinct difference in heat effect between wet-moulded and dry-pressed material. The cast body reached a given degree of porosity (not too far removed from complete vitrification) at the lowest temperature, and the dry-pressed material at higher temperatures, according to the pressure applied. Minimum porosity was reached simultaneously in all cases except by the pieces pressed at 2000 lb. per sq. in., which showed a slight lag. Complete vitrification was not reached at  $1325^\circ\text{C}$ . by the plastic-moulded sample or the one dry-pressed at 2000 lb. per sq. in. The relative compactness of a porcelain body appears to govern the temperature at which complete vitrification takes place and the time during which a given temperature must be maintained.—A. B. S.

*Testing furnace [for ceramic products]; Construction of a novel —.* S. F. Walton. J. Amer. Ceram. Soc., 1920, 3, 833—836.

A SIMPLE form of gas-fired furnace for use in testing the modulus of rupture of refractory materials at high temperatures consists of a square base with well-hole and main flue, surmounted by a cylindrical wall built of carborundum bricks backed by  $1\frac{1}{4}$  in. of rammed kieselguhr and  $\frac{1}{4}$  in. of fireclay bricks. The gas-ports were of galvanised iron, surrounded by fireclay and set tangential to the wall. The main flue was made by placing a 5-in. galvanised iron pipe inside a 7-in. iron pipe and filling the annular space with refractory cement. As the metal burned away it left the refractory material firm. For making the cross-breaking tests a lever arrangement is used, one end of the lever pressing through a knife edge of refractory material on the test-piece, which rests on knife-edges embedded in the floor of the furnace. The other end of the lever is connected with a chain jack, having a handle fitted to the sprocket wheel and set on a platform scale. A bucket hung from the balance arm of the scale is loaded by water from a Mariotte bottle.—A. B. S.

*Dolomite.* Stull. See VII.

#### PATENTS.

*Bores in glass; Production of —.* J. Kent. E.P. 153,824, 28.2.20.

GLASS tubing having more than one bore is made by forming an approximately cylindrical ball of glass on a single blowing-iron. A sealed length of exhausted glass tubing extending the length of the ball is autogenously united with it and the piece thus constituted is brought to the desired cross-sectional shape and drawn out to the required dimensions. By bedding-down a length of glass rod of lenticular cross-section upon the surface of the piece immediately over the small diameter bore and drawing out, a lens-fronted cylindrical bore thermometer tube with a capillary bore in the thickness of its wall is obtained.—H. S. H.

*Flake materials [mica]; Manufacture of sheets by aggregation of —.* The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 153,612, 25.2.18.

FLAKES (e.g. of mica) are deposited by a current of air upon a perforated endless belt travelling upon a horizontal rotating openwork cylinder within which suction is created. The distribution of flakes is rendered more uniform by obstructing the air current by a subsidiary screen. The air-current first strikes the fresh portion of the travelling screen and there deposits the larger flakes. The swirl of left-over flakes remains sufficiently long in contact with the screen and the initially formed layer to cover the small openings in the layer with flakes. The layer is carried round until it becomes horizontal, then receives adhesive and is manifolded.—H. S. H.

*Clay; Treatment of —.* P. Schidrowitz, W. Feldenheimer, and W. W. Plowman. E.P. 153,961, 2.6.19.

CLAY, preferably purified, is suspended in water by means of a deflocculator (e.g. sodium carbonate), and before any settlement of clay substance takes place the whole clay suspension is evaporated to dryness. The suspension is made either by mixing the clay with sufficient water to produce a pasty mass and adding the deflocculator in the required quantity to obtain fluidity, or by directly working up the clay with water and the deflocculator together. Clay dried in a deflocculated state possesses qualities which are not obtainable when the drying is effected immediately after flocculation, and is suitable, for example, for incorporating in rubber mixings (cf. E.P. 153,343; page 19 A). —H. S. H.

*Abrasive block.* F. P. Jones and A. Fleming. E.P. 153,692, 18.8.19.

SILICA sand, firestone, glass, or china (76 pts. by weight), magnesia (7), magnesium chloride (7), wax, sawdust, sulphur, or ammonium chloride (10), and water (10) are mixed together and moulded into blocks. When set the material is heated until the wax or its substitute is removed, the porosity of the block depending on the amount of wax or the like used.—H. S. H.

*Furnace for melting glass and other purposes.* J. S. Shaw. U.S.P. 1,359,756, 23.11.20. Appl., 4.5.20. SEE E.P. 144,744 of 1918; J., 1920, 573 A.

## IX.—BUILDING MATERIALS.

*Portland cement; Analysis of —.* F. Ferrari. Giorn. Chim. Ind. Appl., 1920, 2, 434—437.

A RAPID and exact method is described by which the following three groups of determinations are carried out simultaneously: (1) moisture; (2) loss on calcination, silicic acid, alumina, lime, and magnesia; (3) insoluble silicious residue, ferric oxide, and sulphuric acid. The method used for the rapid estimation of the silicic acid is based on the practically complete insolubility of gelatinous silica with a low water-content, and the iron is estimated by means of nitrosophenylhydroxylamine-ammonium (cupferron). The procedure to be followed is described in detail. The method is applicable to the analysis of limestone, unburnt cement, clays, etc., if these are subjected to preliminary scorification at about 1400° C.—T. H. P.

*Iron-Portland cement and puzzuolana; Mixtures of —.* F. Ferrari. Giorn. Chim. Ind. Appl., 1920, 2, 549—554. (Cf. J., 1920, 821 A).

CEMENTS composed of mixtures of normal Portland cement and puzzuolana, although when set offering

a much greater resistance than pure Portland cement to the action of sulphates and chlorides, eventually undergo slow disintegration, like all mixtures containing strongly basic aluminates. Normal mixtures with a base of iron Portland cement undergo retrogradation after a long period owing to the diffusion of the lime, whereas mixtures of iron Portland cement and puzzuolana are quite stable and practically impermeable and are suitable for use in contact with sea-water or water containing sulphates.—C. A. M.

*Plaster; Dispersoid and colloid chemistry of —.* Wo. Ostwald and P. Wolski. Kolloid Zeits., 1920, 27, 78—92.

THE rate of setting of plaster and the processes occurring therein have been investigated by a study of the change in the viscosity of 3—5% suspensions of burnt gypsum. Such suspensions show an increase in viscosity for the first half-hour after preparation. The time-viscosity curves are S-shaped. The concentration of plaster has a marked influence on the setting, since the difference between the initial and final viscosities (hydraulic effect) increases extraordinarily rapidly with the concentration. No setting takes place at 60° C. and very little at 37° C., but at 0° C. the increase in viscosity, that is the setting, is very great. Freshly ground preparations show a greater hydraulic effect than aged specimens. The more finely divided the plaster the greater the hydraulic effect. The addition of potassium chloride up to 0.2N accelerates the setting of plaster, but eventually gives a smaller hydraulic effect than pure water. Concentrated solutions of potassium chloride retard the setting. Acetic acid, 2N, retards the setting but gives a somewhat larger hydraulic effect. Gelatin, 0.2%, and alcohol, 20%, completely inhibit the setting.—J. F. S.

*Wooden poles and sleepers; Zinc fluoride as a preservative for —.* R. Nowotny. Oesterr. Chem.-Zeit., 1920, 23, 136—138.

Tests on 15,763 telegraph poles extending over eight years show that zinc fluoride is a particularly suitable preservative, as it is soluble with difficulty and highly toxic to mould growths. The wood is soaked in a mixture of zinc chloride and sodium fluoride solutions, each of 1.75% concentration, and then heated to 45°—60° C. to form zinc fluoride or the basic fluoride. The total wastage of poles in eight years was only 6% (the loss for any one year never exceeding 1.82%), as compared with a total of 31% in the case of similar poles treated with copper sulphate. The best results are obtained when the timber absorbs about 3 kg. of zinc fluoride per ch. m. The chief drawbacks to the use of zinc fluoride are the inconvenience of liquid zinc chloride in transport and the premature formation of the fluoride. The latter may be prevented by neutralising the sodium fluoride solution by adding sulphuric acid or sodium bisulphate to it (the mixture must not react acid to methyl orange) prior to adding it to the zinc chloride. The use of zinc sulphate instead of zinc chloride (Renfer & Co. A.-G., Swiss Pat. 75,478 of 1917) is preferable. A satisfactory preservative may also be made by mixing powdered zinc sulphate, sodium sulphate, and sodium bisulphate in suitable proportions and dissolving the mixture in water when required for use. The timber should be thoroughly dried prior to impregnation.—A. B. S.

## PATENTS.

*Rotary kiln.* A. Larsen, Assr. to F. L. Smidth & Co. U.S.P. (A—C) 1,358,759—61, 16.11.20. Appl., 12.5.20.

(A) A ROTARY kiln is divided into upper and lower kiln portions by a chamber communicating with

both portions and containing a partition wall with damper between the ends of the two portions of the kiln, so that products of combustion from one side of the partition can be directed to a heating chamber and thence to the other side of the partition. Means are provided to convey material from the upper portion of the kiln to the lower. (a) A rotary kiln comprises a continuous rotary shell with an opening through it and a casing surrounding the shell in line with the opening. Means are provided for conducting the products of combustion delivered to the casing through the opening in the shell to a heating chamber. Raw material is conducted within the shell from a point above the opening through the shell to a point below the opening. (c) A primary rotary kiln adapted to receive an excess of slurry in its evaporating and drying zone is provided between the evaporating and heating zone and the zone in which carbon dioxide is driven off, with an opening through which the excess of dried raw meal may escape and be transferred to a secondary sintering kiln.—H. S. H.

*Concrete structures; Building material for —.*  
F. L. Smith & Co. E.P. 137,330, 2.1.20. Conv., 1.5.18.

GROUND "moler" (diatomaceous earth) is heated until sintering starts (about 1100° C.) and mixed with Portland cement. The resulting concrete possesses greater elasticity and is a better insulator against heat and sound than concrete prepared with sand or gravel.—H. S. H.

*Impregnating wood to preserve it; Process for —.*  
Grubenholz - Imprägnierung G.m.b.H. E.P. 141,728, 14.4.20. Conv., 29.11.13.

THE addition of an alkali chromate, bichromate, bromate, iodate, or chlorate, borax, or a dialkali phosphate, or a mixture of the same, to a solution of dinitrophenol or its salts for impregnating wood prevents decomposition of the solution when in contact with iron, and thus permits the impregnation to be carried out in iron vessels.—L. A. C.

*Wood; Preservation of —.* Deuts. Erdöl-A.G., and F. Seidenschnur. G.P. 325,543, 9.8.17.

WASTE material obtained from the wood, such as bark, sawdust, etc., is distilled, the waste heat from the retorts is employed for drying the wood, and the products of the distillation are used as a preservative.—L. A. C.

*Wood; Process of and means for effecting colour transformation or change in — and apparatus therefor.* F. E. Williams. E.P. 153,619, 12.6.19.

WOOD is immersed in a boiling aqueous solution of copper sulphate and ferrous sulphate in substantially equal proportions.—H. S. H.

*Sound-absorbing material for walls and ceilings.*  
W. C. Sabine (J. D. K. Sabine, extrix.) and R. Gutavino. Reissue 14,992, 23.11.20, of U.S.P. 1,197,956, 12.9.16. Appl., 24.7.18.

SEE E.P. 110,194 of 1916; J., 1917, 1237.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Iron in basic open hearth [steel] practice; Use of high-manganese —.* E. A. Wheaton. Amer. Iron and Steel Inst., Oct., 1920. Blast Furnace and Steel Plant, 1920, 8, 596—598.

Pig iron containing 1.39—2.13% Mn manufactured in the blast furnace with lean slags and having a sulphur content as high as 0.1% was de-

livered to the open-hearth furnace through a mixer with a 50% elimination of the sulphur. This iron improved the quality of the steel, the manganese diminishing the sulphur in the charge and eliminating the oxygen. The tonnage was maintained, and the average time of heats showed a considerable reduction, possibly due to the quicker working of the slags. There was no excessive scorification of furnace bottoms or banks, nor did the iron have a detrimental effect on ladle linings. The high-manganese iron is of great assistance in the manufacture of high-grade and alloy steels, and steels made from it show increased yields with ordinary rolling mill practice.—J. W. D.

*Manganese in the basic open-hearth steel process; Utilisation of —.* E. Killing. Stahl u. Eisen, 1920, 40, 1545—1547.

MANGANESE is utilised most efficiently when the charge is tapped when the manganese reduced from the slag is at a maximum; when bases are added in quantities corresponding with the absorption capacity of the slag, avoiding excess or deficiency; when the charge has the smallest possible content of acid elements such as phosphorus and silicon, so that large slag volume is avoided; when the temperature is as high as possible, and when manganese is added in the metallic state.—J. W. D.

*Iron-carbon alloys; Graphitisation in —.* K. Honda and T. Murakami. Iron and Steel Inst., Sept., 1920. 8 pages. [Advance proof.]

FROM experiments with a pure white iron free from graphite, melted in 20-g. lots in an electric furnace, it is considered that graphitisation is the result of decomposition of solid Fe<sub>3</sub>C, and that graphite does not separate directly from the liquid phase. The reaction is a catalytic one due to carbon monoxide or dioxide. At the high temperatures carbon monoxide is in part converted into carbon dioxide and carbon. The dioxide then reacts with Fe<sub>3</sub>C as follows: CO<sub>2</sub> + Fe<sub>3</sub>C = 2CO + 3Fe. Carbon dioxide is again formed from the monoxide, and the Fe<sub>3</sub>C is thus converted gradually into iron and graphite. A high melting temperature of 1300° C. or over hinders the formation of graphite. A sample melted at 1200° C., cooled to 1130° C., and quenched shows no graphite. If, however, the quenching temperature is lowered to 1050° C., full graphitisation occurs. If carbon monoxide or dioxide is passed through the melt, and the metal immediately cooled, a high degree of graphitisation is found. Air or iron oxide also favours the formation of graphite. Hydrogen or nitrogen produces no graphitisation. In alloys melted in *vacuo* graphite does not readily form.—F. C. Th.

*Spheroidal cementite [in steel]; Formation of —.* K. Honda and S. Saito. Iron and Steel Inst., Sept., 1920. 7 pages. [Advance proof.]

IF a quenched carbon steel is reheated to a temperature below the Acl point the carbide of the sorbito becomes spheroidal. In hypereutectoid steels reheating to a temperature between Acl and that at which all the carbide passes into solution results in the formation of spheroidal cementite. Lamellar pearlite does not give spheroidal cementite when heated below the Acl point, but at or just above that temperature it becomes globular. Granular pearlite, however, becomes globular when heated below the Acl temperature. In low carbon steels the temperature interval in which formation of spheroidal cementite occurs is 730°—750° C. With higher carbon content the range widens rapidly, and with 1.6% carbon or over the range is 730°—850° C.—F. C. Th.

*Steels; Rates of reaction in certain* — at 930° C.  
E. D. Campbell and B. A. Soule. *Iron and Steel Inst.*, Sept., 1920. 6 pages. [Advance proof.]

BARS of steel 6 mm. square by 15 cm. long were quenched vertically in water from 930° C. They were then examined by means of measurements of thermo-electric potential against electrolytic iron from end to end to determine what differences are induced by the different conditions of cooling. In all freshly quenched steels the end which entered the water last had as a result of the more rapid cooling the higher thermo-electromotive potential. In a pure carbon steel (C 1.18%) the difference would indicate a change in constitution between the two ends equal to 8% of that due to tempering at 110° C. and 3% of that due to the total carbide concentration. Where silicon, nickel, or manganese was present the thermo-electric potential was higher than that due to carbides alone, and tempering at 110° C. resulted in an increase.—F. C. Th.

*Chrome-nickel steels; Non-rusting* — B. Strauss. *Naturwiss.*, 1920, 8, 812—814. *Chem. Zentr.*, 1920, 91, IV., 621.

CHROME steels show great resistance to corrosion, but without nickel have not the requisite mechanical properties. There are two groups of chrome-nickel steels, showing high resistance to corrosion but differing essentially in physical and chemical properties and in structure. To the first group belong steels having a martensitic structure and containing 10–15% Cr with 1–3% Ni. These are self-hardening. By heating slightly above the transition temperature, 680°–750° C., and cooling slowly or quickly, they assume a troostoritic structure and become workable. The second group of chrome-nickel steels, with 20–23% Cr and 6–9% Ni, have a polyhedral structure (austenite) and require a different heat treatment, consisting in heating to 1100°–1200° C. and cooling quickly. Unlike those of the first group, these steels are non-magnetic. In both groups the carbon content is 0.1–0.3%. Between the two groups lie steels with intermediate structure and unfavourable mechanical properties. The oxidation potential of these non-rusting steels is between that of copper and that of silver.—E. H. R.

*Pyrites cinder; Desulphurising of* — F. Fichter and E. Schaffner. *Helv. Chim. Acta*, 1920, 3, 869–872.

SECONDARY roasting for the removal of sulphur from pyrites cinder is ineffective below 1200° C., but reduction in an atmosphere of hydrogen at 1030° C. gives better results. In fact, pyrites and ferrous sulphide themselves may be reduced to iron in this way. Iron almost free from sulphur is also obtained by electrolytic deposition from a solution of pyrites cinder in very concentrated sodium hydroxide (*cf.* J., 1918, 659 A) or by heating the cinder with lime, kaolin, and charcoal in the electric arc.—J. C. W.

*Alloys of aluminium and magnesium*. D. Hanson and M. L. V. Gayler. *Inst. of Metals*, Sept., 1920. [Advance copy.] 27 pages.

A RE-DETERMINATION of the thermal equilibrium diagram of this series has been made. The existence of two compounds is indicated, Al<sub>2</sub>Mg<sub>3</sub> and Al<sub>3</sub>Mg<sub>2</sub>, melting at 450° C. and 455° C. respectively. The latter compound does not occur exactly at a maximum on the liquids. Three eutectics are present, namely between the  $\alpha$ -solution and a solution rich in Al<sub>2</sub>Mg<sub>3</sub>, melting at 447° C. and extending from 13 to 37% Mg; between solutions rich in the compounds, melting at the same temperature, the horizontal extending from 38.5 to 42% Mg; and between the solution rich in Al<sub>2</sub>Mg<sub>3</sub> and the  $\beta$ -solution rich in magnesium, melting at 436° C.

and extending from 60 to 88% Mg. The solid solution of Al<sub>2</sub>Mg<sub>3</sub> in aluminium contains 12% Mg at 447° C. and 10% at room temperatures. The  $\beta$  solution extends from 36.7 to 38.5% Mg at temperatures below 447° C. The  $\gamma$  solution contains 42–59% Mg at 444° C. and 49–60% at 400° C. and at room temperature. At 486° C. magnesium holds 10% Al as Al<sub>2</sub>Mg<sub>3</sub> in solution and at room temperature 9%.—F. C. Th.

*[Aluminium] alloys; Light* — of high resistance. *Grard. Rev. Mét.*, 1920, 17, 286–300.

TEST-PIECES of duralumin annealed at 450° C. gave the following results when tested in the longitudinal and transverse directions respectively: maximum stress, 32, 26 kg. per sq. mm.; elastic limit, 13, 12 kg. per sq. mm.; elongation, 18, 10%; impact test, 3, 2.5 kg.-m. On cold rolling the maximum stress fell to a minimum with between 15 and 20% reduction, after which it again rose slightly. The minimum value was 22 kg. per sq. mm. both longitudinally and transversely. The elastic limit is raised by working, and with 20% or greater reduction nearly coincides with the tensile strength. The elongation is lowered rapidly to about 4% with 20% reduction, and then remains stationary up to 40% reduction. The resilience (impact value) falls to about 1 kg.-m. with 50% reduction. 50% reduction is the practical limit, further rolling leading to cracking. The material reduced 50% by rolling was used for the tests on the influence of annealing after cold work. The tests were carried out after ageing for 8 days. There are two important temperature ranges, viz., 350°–375° C. and 475°–500° C., and the properties of the alloy annealed at each, taken in the direction of rolling, are:

	Elastic limit, kg. per sq. mm.	Maximum stress, kg. per sq. mm.	Elongation, %	Impact, kg.-m.
350° C., slowly cooled ..	6	20	20	6
.. air cooled ..	7	20	20	4.5
475° C., slowly cooled ..	12	28	16	4
.. air cooled ..	18	32	18	4

There are maxima in the elongation and resilience curves at 350° C. and 475° C., minima in the tensile strength and elastic limit curves at 350° C., and maxima of strength and elastic limit at 475° C. 350° C. is the best temperature for softening for machining. When the cold-worked alloy is quenched from 350° and 475° C. respectively, the mechanical properties are: elastic limit, 9, 20 kg. per sq. mm.; maximum stress, 20, 40 kg. per sq. mm.; elongation, 15, 20%; impact test, 3, 3.5 kg.-m.; 475° C. is the best quenching temperature for the final treatment. Quenched at 550° C. the alloy is softer, less ductile, and more brittle than when quenched at 475° C. After quenching at this latter temperature the material hardens by ageing fairly rapidly during the first 4 days, after which it becomes fairly steady. In this period of 4 days the maximum stress rises from 30 to 38 kg. per sq. mm., and the elastic limit from 10 to 23 kg. per sq. mm. The elongation and the resilience show considerable variations during the first 4 days, but after 8 days have about the same values as before ageing. Ageing is best effected by re-heating to 200° C. after quenching from 475° C. in water. The Brinell hardnesses (load 500 kg.) at various temperatures of the quenched and aged alloy are: ordinary temperature, 83; 100° C., 108; 200° C., 83; 300° C., 50; 400° C., 12.—F. C. Th.

*Aluminium; Nickel-plating of* — A. Mazuir. *Ann. Chim. Analyt.*, 1920, 2, 335–336.

THE aluminium is immersed for 2 mins. in a cold bath containing 8 g. Na<sub>2</sub>O and 30 g. KCN per l., and

then thoroughly washed with water and scoured with milk of lime ( $\text{CaO} \cdot 4\text{H}_2\text{O}$ ). After thorough washing it is immersed in a bath of a metal chloride, preferably manganese or iron chloride. A suitable bath contains 350 c.c. of hydrochloric acid of 22° B. (sp. gr. 1.18), manganese 2 g., and water 650 c.c., whilst a ferric chloride bath should contain 3 g. Fe per l. Finally the object is immersed in a bath of nickel sulphate (e.g., 120 g. nickel sulphate and 50 g. of a double nickel sulphate), and electrolytically coated by means of a current within the limits of 2.5 and 3 volts, and ampérage of 1 to 1.5 per sq. dm. The anodes are of thin sheet nickel plate. Plating is complete in 1½ hrs., and the aluminium is then washed with boiling water, and dried by means of compressed air.—C. A. M.

*Zinc; Experimental furnace for the preparation of — and the determination of the various products.* O. Mühlhaeuser. Metall u. Erz, 1920, 17, 487—494.

MINUTE details accompanied by the necessary drawings are given of a small experimental plant for the distillation of zinc and the collection and treatment of the various products. The plant consists of a gas producer, retort, and combustion chamber, a condenser for the zinc, and scrubbing towers to remove the fume from the issuing gases. Exact instructions are given for working the furnace, together with three examples showing the results obtained from charges of ore, crude zinc (90.6% Zn), and zinc dust.—A. R. P.

*Babbitt metal; Analysis of —.* H. C. Boehmer, J. R. Gordon, and C. W. Simmons. Canad. Chem. J., 1920, 4, 171—173.

ONE g. of filings is dissolved in 35 c.c. of strong sulphuric acid. After cooling, 5 c.c. of strong hydrochloric acid and 150 c.c. of water are added and, after standing for 1 hr., the lead sulphate is collected in a Gooch crucible, washed with dilute sulphuric acid, then with water, dried, ignited at a dull red heat for ½ hr., and weighed. The antimony in the filtrate is determined by titration with potassium bromate, using methyl orange as indicator. The titrated solution is reduced by boiling for 20 min. with 0.5 g. of antimony and 40 c.c. of strong hydrochloric acid, cooled in a current of carbon dioxide, and the tin titrated with the same bromate solution, using potassium iodide and starch as indicator. The reduction may also be effected by adding 9 g. of zinc and 40 c.c. of hydrochloric acid, 5 c.c. at a time, while passing carbon dioxide. When all the metal is dissolved, the solution is titrated as before.—A. R. P.

*Metallurgical products not attacked by acids; Rapid method of disintegrating — for analysis.* I. Compagno. Giorn. Chim. Ind. Appl., 1920, 2, 554—555.

ALLOYS of iron with silicon, tungsten, chromium, etc., which are not attacked by mineral acids may be rapidly disintegrated by fusing the finely-powdered metal in a nickel crucible with a dry mixture of sodium carbonate (52%), potassium nitrate (20%), and coarsely-powdered magnesium (28%). When cold, the fused mass is acidified with a mineral acid and the solution boiled until gas ceases to be evolved.—C. A. M.

*Malleability of metals and alloys; New method for determining —.* P. Ludwik. Stahl u. Eisen, 1920, 40, 1547—1551.

To obtain an approximate indication of the toughness and ductility of metals without preparing a special test-piece, a hardened cone with an angle of 90° at the point, such as is used in the Ludwik hardness test, is driven normally into the material under test near its edge until a crack is produced, the edge of the material being bulged outwards a

distance,  $a$ . On account of the geometrical similarity of the mechanism of flow for different distances,  $k$ , of the point of the cone (before the test) from the edge, the ratio  $a/k$  is an approximate index of the malleability of the material in question independent of the actual value of  $k$ . This index can be converted into other useful comparative numbers.—J. W. D.

*Impact tests [on metals].* A. Ono. Mem. Coll. Eng., Kyushu Imp. Univ., 1920, 2, 89—116.

AN error of  $\pm 0.1$  mm. in the diameter of the hole (4 mm. diameter) drilled in certain types of test-pieces for impact tests may give rise to an error of  $\pm 1.5\%$  in the result, which is insignificant. In steels with a banded structure marked irregularity occurs in impact test results. In one case four times as much work was required to break the piece as in other pieces of the same steel. Transverse tests were in general distinctly worse than longitudinal ones. Cold straining of mild steel lowers the resistance to impact, which is restored by annealing. In two cases examined, annealing at 770° C. and at 600° C. respectively was sufficient to restore the toughness. In repeated impact tests the effect of cold straining was not very marked. The effect of grain growth, however, is marked, as illustrated in the following tests on a steel containing 0.065% carbon. After annealing at 920° C. the steel withstood 1010 blows; after annealing, followed by straining (% strain=8%), 940; after annealing, straining, and again annealing at 450°, 500°, or 600° C., 1013; 700° C., 496; 800° C., 553; 900° C., 1104; 980° C., 572 blows.—F. C. Th.

*Arsenic.* Jones. See VII.

#### PATENTS.

*Wrought iron; Manufacture of —.* E. E. Elliott. E.P. 153,523, 15.3.20.

A CHARGE of pig iron or cast iron, together with iron ore, purple ore, or other suitable ore, and tap cinder, is heated in a furnace in a receptacle having an open joint between the sides and bottom, and one or more joints in the sides through which the gases and impurities escape.—J. W. D.

*Steel; Magnetic apparatus for heat treatment of —.* C. O. Bastian. E.P. 153,336, 25.3.19.

THE claim is for a method of determining the critical temperature of steel by heating it in a furnace surrounded by a magnetising coil, the change being indicated on a magnetic compass.

—J. W. D.

*Steel; Heat treatment of —.* C. P. Sandberg and J. C. W. Humfrey. E.P. 153,750, 20.10.19.

STEEL cooling from a temperature above its critical range is brought under magnetic influence, so that as soon as it has cooled to a temperature at which it acquires magnetic properties it becomes part of a magnetic field and attracts a magnet swinging close by, thus giving visual indication for controlling the cooling operation.—J. W. D.

*Manganese steel; Process of making —.* W. G. Nichols, Assr. to American Manganese Steel Co. U.S.P. 1,356,551, 26.10.20. Appl., 21.6.20.

MANGANESE steel scrap is melted, and manganese ore is added in sufficient quantity to restore to the ultimate bath the desired proportion of manganese.

—J. W. D.

*Manganese steel; Making —.* W. G. Nichols, Assr. to American Manganese Steel Co. U.S.P. 1,359,268, 16.11.20. Appl., 15.8.18.

MANGANESE steel is recovered from scrap by melting ordinary steel scrap in an electric furnace, adding to the molten bath all the manganese steel scrap in



one charge, heating the bath by slow stages to the melting point of manganese steel, and adding ferromanganese in sufficient quantity to produce the desired alloy.—J. W. D.

*Iron or steel article; Alloy-coated —, and method of coating same.* J. L. Schueler, Assr. to Keystone Steel and Wire Co. U.S.P. 1,357,907, 2.11.20. Appl., 15.7.16.

IRON or steel wire is drawn through a molten bath consisting of 91% of lead, 8% of antimony, and 1% of zinc, being removed in a vertical direction without wiping, so that excess metal on the wire drains back into the bath.—T. H. Bu.

*Iron-silicon alloy; Process of treating —.* W. J. Beck and J. A. Aupperle, Assrs. to The American Rolling Mill Co. U.S.P. 1,358,408, 9.11.20. Appl., 19.3.19.

ANNEALED sheets of iron-silicon alloy are pickled so as to leave a coating of silicious compounds, and then cold rolled to impregnate the surface with these compounds.—T. H. Bu.

*Steel; Case-hardening —.* J. D. Cutter, Assr. to Climax Molybdenum Co. U.S.P. 1,358,831, 16.11.20. Appl., 28.6.20.

A LOW-CARBON steel containing Ni, Cr, and Mo, each not exceeding 1%.—B. M. V.

*Steel; Process of treating —.* N. C. Einwechter. U.S.P. 1,359,238, 16.11.20. Appl., 5.5.19.

VANADIUM steel is heated slowly to 1430°—1440° F. (about 775°—780° C.), quenched in water at 60° F. (15° C.) until vibration ceases, and then placed in an oil bath.—J. W. D.

*Iron plate; Process of removing scale from —. Method of simultaneously annealing and descaling tin-plate.* I. M. Scott. U.S.P. (a) 1,359,281 and (b) 1,359,282, 16.11.20. Appl., (a) 23.4.20 and (b) 30.6.20.

(A) THE plates are heated in contact with powdered ferrosilicon at a sufficiently high temperature to reduce the scale to metal. (B) The plates are packed in annealing boxes with carbonaceous material between them and heated to a temperature sufficient to reduce the scale and anneal the plates without carbonising them to any considerable extent.—A. R. P.

*Ferro-chromium; Process for increasing the yield of chromium in the aluminothermic production of carbon-free — or multiple alloys thereof containing chromium.* T. Goldschmidt, A.-G. E.P. 152,990, 29.9.20. Conv., 14.10.19.

A CERTAIN amount of iron oxide, e.g., rolling-mill scale, is added to the charge of aluminium and chrome-iron ore before igniting. In the manufacture of multiple alloys of ferro-chromium, oxides of other metals, such as cobalt, nickel, tungsten, molybdenum, or vanadium, are added to the charge.—A. R. P.

*Bearing metals.* Metallbank u. Metallurgische Ges. A.-G. E.P. 140,790, 24.3.20. Conv., 18.1.17.

ALLOYS suitable for bearing metals consist essentially of lead and not more than 5% of barium, and may contain in addition light metals, especially sodium, calcium, and magnesium, and aluminium or small quantities of heavy metals such as copper, zinc, tin, manganese, etc.—J. W. D.

*Magnetic separators.* F. Krupp A.-G. Grusonwerk. E.P. 145,442, 18.6.20. Conv., 2.8.18.

EACH magnet of a magnetic separator of the type in which two or more magnetic fields operate one behind the other, is provided with a separate supply device, capable of being inclined at any angle to

the magnet and independently movable in a vertical direction towards or away from it. The discharge ends of the supply devices are curved so as to be always parallel to the edge of the rotating magnetic annulus, and the whole apparatus is mounted on a pivot in order that the speed of the material may be simultaneously accelerated or retarded for all the magnets.—A. R. P.

*Ores and the like; Process and apparatus for washing —.* C. A. Edgley. E.P. 153,503, 3.2.20.

THE ore, e.g., chloridised roasted ore, is admitted to the centre of a flat-bottomed circular basin having a rotary stirrer, and from the side of which extends a discharge trough with bottom inclined slightly upwards, containing a conveyor. Water or other suitable liquid is allowed to flow into the discharge trough towards the basin, in the opposite direction to the conveyor, and after it has washed the ore, it flows out of the circular basin. The ore is discharged by the conveyor to a similar unit if required.—T. H. Bu.

*Aluminium alloys.* H. C. Hall, and Rolls-Royce, Ltd. E.P. 153,514, 25.2.20.

AN aluminium alloy contains Cu 0.1—3.0%, Ti 0.1—2%, Zn 6.0—16.0%, Fe not exceeding 0.6%, Si not exceeding 0.4%, other elements not exceeding 0.4%. Part of the aluminium, not exceeding 3%, may be replaced by not more than 1.5% of either Sb or Mg, or not more than 1.5% Sb and 1.5% Mg.—J. W. D.

*Aluminium alloys.* H. C. Hall, and Rolls-Royce, Ltd. E.P. 153,823, 25.2.20.

CLAIM is made to aluminium alloys containing either Cu 4—12%, Ti 0.12—2.2%, Fe not exceeding 0.6%, Si not exceeding 0.4%, other elements not exceeding 0.5%, or an alloy of similar composition in which not exceeding 3% of the aluminium is replaced by not exceeding 1.5% of antimony and/or not exceeding 1.5% of magnesium.—J. W. D.

*Alloy.* F. E. Carter, Assr. to Baker and Co., Inc. U.S.P. 1,355,811, 19.10.20. Appl., 15.2.19.

AN alloy containing 83.3% Au and 16.7% Ni has the colour of, and is capable of being worked in the same way as, platinum.—J. W. D.

*Alloy.* F. E. Carter, Assr. to Baker and Co., Inc. U.S.P. 1,357,272, 2.11.20. Appl., 20.1.20.

THE alloy consists of platinum with 5—15% Au, 0.5—5.0% Ag, and 0.5—5.0% Pd.—A. R. P.

[Alloy for] apparatus for high temperature uses. Chemical ware or technical apparatus and alloys for making same. F. A. Fahrenwald. U.S.P. (a) 1,357,549 and (b) 1,357,550, 2.11.20. Appl., 25.8.19.

(A) AN alloy of iron with 10—20% each of cobalt and chromium, and at least 0.2% each of carbon, silicon, and manganese. (B) The alloy contains 40—60% Cr, together with 10—20% of one or more metals of the chromium group, alloyed with one or more iron group metals.—A. R. P.

*Zinc alloy.* H. Goldschmidt and K. Müller, Assrs. to The Chemical Foundation, Inc. U.S.P. 1,358,147, 9.11.20. Appl., 19.4.17.

THE alloy contains 0.5—6% of aluminium and 0.25—2% of manganese.—T. H. Bu.

*Alloy; Antifriction — and method of manufacture.* L. M. Parkhurst, Assr. to H. Kramer and Co. U.S.P. 1,359,191, 16.11.20. Appl., 8.3.20.

AN alloy containing approximately Pb 79%, Sb 12%, Sn 6%, Cu 2.5%, and P 0.5%.—J. W. D.

*Corrosion; Prevention of —.* M. A. Adam. E.P. 153,616, 13.5.19.

IRON apparatus, e.g., a tank, pump, or pipe, carry-



ing corrosive solutions of metals which are capable of being deposited electrolytically, is made the cathode in an electric circuit, the anode being, preferably, carbon, and a current is passed through the solution at such a potential that it deposits a metallic protective coating on the iron at a speed at least as rapid as that at which the solution redissolves it. A heater for such corrosive solutions consists of a water-jacketed iron vessel containing an anode, preferably carbon, arranged to maintain contact with the solution throughout its passage through the vessel. Hot water is circulated through the jacket. The invention may be applied to the protection of iron vessels for use in detinning tin-plate scrap.—A. R. P.

*Briquetting ores.* W. Tyrrell, Assr. to New Era Iron and Steel Corp. U.S.P. 1,356,100, 19.10.20. Appl., 29.5.19.

ORE briquettes are made with a binding material having a higher fusing point than the metals in the ores, the composition of the binding material by weight being sodium sulphate 2 pts., diatomaceous earth 2, concentrated lye 2, magnesium carbonate 1 pt.—J. W. D.

*Smelting furnace for lead ores and the like.* J. Labarthe. U.S.P. 1,356,159, 19.10.20. Appl., 30.9.19.

AN elongated reduction furnace has an opening at the top extending the length of the furnace, and closed by a sliding door. Below the opening are vents for fumes and gases. Ore conveying and dumping compartments, each one-half the length of the furnace, travel on a track adjacent to the opening in the top of the furnace.—J. W. D.

*Furnace; Metallurgical* —. H. B. Bryson. U.S.P. 1,358,703, 16.11.20. Appl., 26.9.17.

IN a process of making steel direct from iron ore, scrap, or the like, the materials are heated in a carbon-lined furnace, the molten metal being covered with a vitreous slag so as to maintain conditions similar to crucible conditions.—J. W. D.

*Annealing metal castings.* D. MacIntosh. U.S.P. 1,356,644, 26.10.20. Appl., 11.7.19.

THE castings are imbedded in a large quantity of granular calcium carbonate, heated to a high temperature, and then gradually cooled.—J. W. D.

*Solder.* B. E. Newell. U.S.P. 1,357,297, 2.11.20. Appl., 8.11.19.

A MIXTURE of 1 pt. of ammonium chloride, 2 of borax, 5 of mercury, 20 of block tin, and 26 pts. of lead.—A. R. P.

[*Copper ores;*] *Metallurgical process [for —].* W. E. Greenawalt. U.S.P. 1,357,495, 2.11.20. Appl., 10.6.18.

COPPER ores are leached with acid, and the resulting rich solution of copper salts is electrolysed to produce metallic copper and to oxidise the salts of metals of a variable valency. The wash liquors from the leaching operation are treated to precipitate copper sulphide, which is mixed with the rich solutions, after first reducing them with sulphur dioxide, and serves to keep them reduced during the electrolysis.—A. R. P.

*Copper ores; Process of treating* —. *Process of treating copper ores with an SO<sub>2</sub> solution for the recovery of the copper therefrom.* N. C. Christensen. U.S.P. (A) 1,357,952 and (S) 1,358,619, 9.11.20. Appl., (A) 30.9.19, (S) 4.4.19.

(A) COPPER is precipitated as a cupro-cupric sulphite by addition of copper to a sulphite solution. (S) Copper ores are lixiviated with a solution containing sulphur dioxide, and copper precipitated as a

cupro-cupric sulphite from the resulting liquor by addition of cuprous oxide.—J. S. G. T.

*Stove for blast-furnaces and gas-producers; Air heating* —. T. Aramaki. U.S.P. 1,357,675, 2.11.20. Appl., 30.4.19.

THE body of the stove is provided with a brick wall having a refractory brick lining on the inner side and a reinforced comparatively non-conducting concrete covering on the outer side.—J. W. D.

*Titanium; Recovery of — from its ores.* O. T. Coffelt. U.S.P. 1,357,690, 2.11.20. Appl., 6.12.18.

TITANIUM ores containing iron are digested with sulphuric acid of 77–85% strength, and the titanium deposited from the solution of iron and titanium sulphates formed.—T. H. Bu.

*Cupola treatment of metals.* H. Koppers, Assr. to Koppers Development Corp. U.S.P. 1,357,780, 2.11.20. Appl., 3.9.20.

A pool of slag is maintained continuously in the hearth of the cupola in the presence of incandescent carbon that has sunk below the level of the air blast. The re-melted iron passes through this pool of slag and gives up sulphur to the slag.—J. W. D.

*Blast treatment of metals.* H. Koppers, Assr. to Koppers Development Corp. U.S.P. 1,357,781, 2.11.20. Appl., 3.9.20.

THE slag and molten metal are continuously maintained at constant levels at the bottom of the charge, so as to prevent sudden sinking of the charge. At the same time a portion of the furnace gas is withdrawn between the top gas outlet and the tuyères so as to check fusion in the stack and to insure gradual sinking of the charge.—J. W. D.

*Crucible treatment of metals.* H. Koppers, Assr. to Koppers Development Corp. U.S.P. 1,357,782, 2.11.20. Appl., 3.9.20.

THE crucible has a foraminous partition dividing it into upper and lower compartments, so that the molten metal charged into the upper compartment flows through the lower compartment in the form of a spray. The slag and molten metal in the lower compartment are maintained at constant levels.—J. W. D.

*Detinning; Method of* —. C. F. Carrier, jun., Assr. to The Vulcan Detinning Co. U.S.P. 1,358,136, 9.11.20. Appl., 5.8.11. Renewed 7.8.20.

SEPARATE portions of tinned scrap are treated in separate successive stages, first with anhydrous chlorine so extremely diluted that reaction with the iron of the scrap is impossible until the greater portion of the tin is removed, and then with concentrated chlorine until detinning is complete.—T. H. Bu.

*Platinum and other metals; Method of extracting — from their ores.* S. S. Sadtler. U.S.P. 1,358,248, 9.11.20. Appl., 8.12.16.

THE ore is mixed with a practically saturated solution containing alkali metal ions to form a pulp through which an electric current is made to pass, while the pulp is agitated to bring its particles into contact with a mercury electrode by which the current is introduced.—T. H. Bu.

*Ores; Process for calcining* —. W. H. Corbould. U.S.P. 1,358,293, 9.11.20. Appl., 7.10.19.

FINELY pulverised copper ores and gangue are heated in a roasting furnace while being subjected alternately to air at a pressure above atmospheric and a partial vacuum.—T. H. Bu.

*Magnetisable material; Process of treating* —. T. D. Jensen, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,358,810, 16.11.20. Appl., 4.4.19.

A METALLIC material is raised to a high tempera-

ture, short of fusion, in an oxidising atmosphere and then slowly cooled, in order to improve its magnetic qualities.—B. M. V.

*Smelting volatile ores; Process and apparatus for* — W. D. Kilbourn, Assr. to United States Smelting, Refining and Mining Co. U.S.P. 1,358,856, 16.11.20. Appl., 14.7.17.

A BLAST furnace for smelting volatile ores comprises a comparatively low shaft having a reducing chamber at its lower end, a grate at the bottom of the reducing chamber for supporting a comparatively low column of ore, into which an air blast is directed through a series of tuyères in the shaft near the bottom of the reducing chamber. A heat-radiating crucible is mounted below the grate. Means are provided for regulating the downward flow of air through the grate, and for removing volatilised oxide from the upper end of the furnace.

—J. S. G. T.

*Metallic articles; Method of producing formed* — C. L. Gebauer, U.S.P. 1,359,353, 16.11.20. Appl., 16.10.18.

A FINELY divided metallic substance is pressed to the desired shape and size under great pressure and then heated to such a temperature that the particles become sintered together without fusion.—A. R. P.

*Vanadium; Process for recovering* — from certain iron ores. W. M. Goodwin and A. F. G. Cadenhead, Assrs. to W. L. Goodwin and W. P. Firth. U.S.P. 1,359,473, 16.11.20. Appl., 4.4.19.

IRON ore containing vanadium is treated in a furnace to produce a pig iron containing all the vanadium and certain other elements in the ore. The pig iron is decarbonised so as to produce a mild steel and a slag containing all the vanadium, and the latter is put through the original process again with a fresh charge of ore. By continuing the process several times it is possible to produce a high-grade ferro-vanadium.—A. R. P.

*Vanadium; Process for recovering* — from certain iron ores. W. M. Goodwin and A. F. G. Cadenhead. E.P. 153,926, 16.5.19.

SEE U.S.P. 1,359,473; preceding.

*Tin; Method of recovering* — from stanniferous waste products. T. A. Eklund. U.S.P. 1,359,494, 23.11.20. Appl., 5.2.20.

SEE G.P. 316,111 of 1919; J., 1920, 303 A.

*Magnesium or alloys thereof and chlorine; Process and apparatus for electrolytic decomposition of anhydrous magnesium chloride and production of* — Production of magnesium or alloys of magnesium and by-products. E. A. Ashcroft. U.S.P. 1,359,653—4, 23.11.20. Appl., 12.8.19.

SEE E.P. 152,402—3 of 1919; J., 1920, 788 A.

*Alloy for cable-casings and the like; Metal* — D. Stenquist. U.S.P. 1,360,045, 23.11.20. Appl., 16.10.19.

SEE E.P. 136,143 of 1919; J., 1920, 602 A.

*Furnaces.* E.P. 153,511. See I.

## XI.—ELECTRO-CHEMISTRY.

*Ferrous and ferric [hydr]oxides; Electrolytic formation of the alkali salts of* — G. Grube and H. Gmelin. Z. Elektrochem., 1920, 26, 459—471. (Cf. J., 1920, 363 A.)

IRON, which has been activated by cathodic treatment, passes into solution in 40% sodium hydroxide in the absence of air, as sodium ferrite, when anodically polarised by a low current density. At

80° C., with a current density of 0.166 amp. per sq. dcm., a 0.026 molecular solution of ferrite may be obtained. Iron may also pass into solution in the hexavalent condition, but not in the trivalent condition in strong alkaline solutions. The electrolytic formation of sodium ferrite is brought about on platinum electrodes either by the anodic oxidation of an alkaline solution of ferrous hydroxide or by the cathodic reduction of a solution of sodium ferrate. (Cf. J.C.S., Jan., 1921.)—J. F. S.

*Townsend cell.* Hooker. See VII.

*Electro-analytical practice.* Böttger. See XXIII.

### PATENTS.

*Electric furnaces; Heating elements for* — E. L. Smalley. E.P. 153,701, 22.8.19.

A REMOVABLE heating element comprises a hollow muffle, the outer surface of which is provided with longitudinal grooves in which lie the parallel strands of a preformed grid resistor of comparatively rigid material. The muffle is shorter than the furnace housing, and the bends of the grid are supported in recesses formed on the end walls of the furnace. Alternatively, the hollow muffle may be provided with end collar portions extending beyond the bends of the grid.—J. S. G. T.

*Furnace; [Protecting resistors of] electric [crucible]* — R. R. Reed. U.S.P. 1,357,901, 2.11.20. Appl., 27.3.20.

DURING the operation of the furnace, the resistors are embedded in a bath of molten glass surrounding the crucible. The crucible and resistors are mounted so as to allow for expansion and contraction of the glass.—J. S. G. T.

*Furnace; [Tilting] electric* — S. N. Castle. U.S.P. 1,358,617, 9.11.20. Appl., 14.6.18.

ALTERNATING current from a transformer is supplied to the electrodes of a tilting electric furnace through leads, the reluctance of which is substantially that of air, and which are supported by the upper parts of projections extending upwards from the furnace structure and tilting therewith.—J. S. G. T.

*Graphite articles [electrodes]; Method of manufacturing* — F. R. Kemmer, Assr. to Republic Carbon Co. U.S.P. 1,357,290, 2.11.20. Appl., 18.12.19.

CARBON articles, such as electrodes, are graphitised in an electric furnace in which the current flows transversely to the largest dimensions of the articles. The articles are arranged in the furnace with their longest dimensions vertical.—J. S. G. T.

*Electrolytic cell.* O. H. Jewell, Assr. to Chlorine Products Co. U.S.P. 1,357,400, 2.11.20. Appl., 4.3.20.

THE cathode of an electrolytic cell comprises a conducting portion, a diaphragm composed of an organic substance resistant to the action of caustic alkali, and porous material protecting the exposed face of the diaphragm from the anolyte.

—J. S. G. T.

*Electrolytic cell and method of using the same [; Cathode for —]* — W. M. Jewell, Assr. to Chlorine Products Co. U.S.P. 1,357,401, 2.11.20. Appl., 21.4.19.

A CATHODE is constituted of a hollow body entirely surrounded by a porous diaphragm in contact with it over its entire area.—J. S. G. T.

*Electrolytic cell.* T. B. Walker. U.S.P. 1,357,485, 2.11.20. Appl., 6.1.17.

THE level of electrolyte in a closed electrode compartment of the cell is controlled by a float operating in a chamber communicating with the elec-

trade compartment by channels respectively above and below the normal level of electrolyte. The movements of the float are transmitted through a liquid seal pipe, one end of which is open to the atmosphere, the other end being sealed by the liquid normally in the float chamber.—J. S. G. T.

Diaphragm for electrolytic cells. M. W. Krejci and G. E. Johnson. U.S.P. 1,358,858, 16.11.20. Appl., 16.1.20.

THE base of the diaphragm is composed of loose filaments of vitrified material compressed so that the diaphragm operates by osmosis.—J. S. G. T.

Electrolytic cell [Diaphragm for —]. B. Thomas. U.S.P. 1,359,002, 16.11.20. Appl., 10.6.18.

A DIAPHRAGM of an electrolytic cell is composed of arenaceous quartz sintered together.—J. S. G. T.

Storage batteries; Separator for —. O. C. Hirsch and A. R. Harnes. U.S.P. 1,358,207, 9.11.20. Appl., 2.1.20.

A WOODEN plate is treated with high-pressure superheated steam and then with an aqueous solution of sulphuric acid to obtain a relatively porous separator for storage batteries.—J. S. G. T.

Depolarising agent for dry batteries, and process of making same. C. Ellis, Assr. to National Carbon Co. U.S.P. 1,359,626, 9.11.20. Appl., 31.10.17.

A DEPOLARISING agent possessing a greater conductivity lag than ordinary pure hydrated manganese dioxide is composed of precipitated slightly hydrated manganese material containing more than 95% of  $MnO_2$ .—J. S. G. T.

[Zinc] battery anodes; Process of making —. H. F. French, Assr. to National Carbon Co., Inc. U.S.P. 1,359,244, 16.11.20. Appl., 27.12.15.

A ZINC electrode is shaped in a mould heated to substantially the melting point of zinc, and the mould is then cooled so that the electrode solidifies slowly.—J. S. G. T.

Electrolytic cells; Indicators ["ionterns"] for —. I. H. Levin. E.P. 154,089, 31.12.19.

Electrical precipitation. U.S.P. 1,357,886. See I.

Gas purification. U.S.P. 1,358,030—2. See I.

Treating fibres. U.S.P. 1,357,580. See V.

## XII.—FATS; OILS; WAXES.

Oils; Polymerisation of —. H. Wolff. Kolloid Zeits., 1920, 27, 183—188.

A THEORETICAL discussion of the processes occurring during the "polymerisation" of linseed oil and tung oil, and the various changes both physical and chemical brought about by the treatment. (Cf. Morrell, J., 1915, 165.)—J. F. S.

Ricinoleic acid; Position of the double bond in —. K. Stosius and K. Wiesler. Biochem. Zeits., 1920, 111, 1—8.

ON oxidising ricinoleic acid with an alkaline solution of potassium permanganate, a mixture of azelaic and suberic acids was obtained. By neutralising the mixture with a known amount of sodium hydroxide and then adding in succession, in four equal portions, the quantity of hydrochloric acid necessary to liberate the free acids and extracting each fraction with ether, azelaic acid, m.p.  $106^{\circ}\text{C}$ ., was obtained in the first fraction, and suberic acid, m.p.  $140^{\circ}\text{C}$ ., in the last fraction. The other fractions contained a mixture of both acids. It is suggested that azelaic and suberic acids are formed by the oxidation of two different isomers of ricinoleic

acid, which arise by displacement of the double bond.—S. S. Z.

Soap; Possibility of substituting inorganic colloids for —. G. Weissenberger. Kolloid Zeits., 1920, 27, 69—78.

AN inorganic substitute for soap may be prepared from a mineral found at Gaura, Siebenbruggen, Austria. The mineral, dried at  $105^{\circ}\text{C}$ ., has the composition:  $\text{SiO}_2$  71.05%,  $\text{Al}_2\text{O}_3$  15.40%,  $\text{Fe}_2\text{O}_3$  1.33%,  $\text{FeO}$  0.21%,  $\text{CaO}$  1.87%,  $\text{MgO}$  0.84%,  $\text{K}_2\text{O}$  3.23%,  $\text{Na}_2\text{O}$  2.67% and  $\text{H}_2\text{O}$  4.18%. When ground finely with water it gives a stable suspension, and on keeping deposits a grey powder, which is colloidal and strongly hydrated and possesses the essential properties of soap.—J. F. S.

Oxidation of paraffin wax. Schaarschmidt and Thiele. See IIa.

Oxidation of paraffin wax. Grün and Wirth. See IIIa.

Oxidation of hydrocarbons. Gränacher. See IIa.

Nutritive fats. Rosenbaum. See XIXa.

### PATENTS.

Oils and the like; Method and apparatus for extracting —. R. Wells, Assr. to Cobwell Corp. U.S.P. 1,357,365, 2.11.20. Appl., 29.2.18.

FOR the extraction of oils or the like from materials containing water, the material is supported in a chamber so as to be exposed above and below, a solvent for the oil, insoluble in water, is run in to an amount sufficient to immerse the material, and the temperature is then raised to a point where the combined vapour pressures of solvent and water are equal to or greater than the vapour pressure in the chamber, more solvent being added as required to maintain the amount in the chamber substantially constant.—A. de W.

[Oxidised] oils; Method of removing or decreasing objectionable odours from treated —. J. E. Booge, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,357,836, 2.11.20. Appl., 13.6.18.

THE objectionable odours from partly oxidised vegetable or animal oils are removed or decreased by blowing the oils with a dry inert gas at  $105^{\circ}$ — $130^{\circ}\text{C}$ .—A. de W.

Cleaning agents; Manufacture of —. F. M. Mayrhofer. E.P. 148,222, 26.8.20. Conv., 27.5.19.

CLEANING agents, which in presence of water yield colloidal materials, are obtained by mixing a dry hydrated or anhydrous, non-hygroscopic, water-soluble aluminium salt, e.g., aluminium sulphate, with a soluble or an insoluble hydrated or anhydrous carbonate, bicarbonate, or percarbonate, e.g., sodium carbonate, bicarbonate, or percarbonate, or magnesium carbonate. A bleaching compound may also be added to the mixture.—A. de W.

Cleaning process. F. Steimmig. G.P. 325,796, 24.5.16.

GELATINOUS silicates are used. A suitable gel is obtained by adding a solution of 24 g. of water-glass of  $38^{\circ}$ — $40^{\circ}\text{B}$ . (sp. gr. 1.36—1.38) in 1 l. of water to a solution of 20 g. of crystallised magnesium chloride in 1 l.—E. H. R.

Fatty and oily matters; Process for the extraction of — from wet sludges and other substances. A. J. Stephens. From Sharples Specialty Co. E.P. 153,668, 11.8.19.

SEE U.S.P. 1,328,047 of 1920; J., 1920, 240a.

Oil filters. J. Potter. E.P. 153,760, 29.10.19.

Cellulose acetate composition. U.S.P. 1,357,335. See V.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Resins; Constituents of* —. *Amyrins from Manila elemi resin and their separation.* A. Zinke, A. Friedrich, and A. Rollett. *Monatsh. Chem.*, 1920, 41, 253—270.

CRUDE amyrin benzoate may be separated, by fractional crystallisation from acetone, into  $\alpha$ - and  $\beta$ -amyrin benzoates, the  $\alpha$ -compound being subsequently purified by alternate treatments with ethyl acetate and acetone. (*Cf. J.C.S., Jan., 1921.*)  
—T. H. P.

*Adsorption compounds of dyestuffs.* Haller. *See IV.*

*Polymerisation of oils.* Wolff. *See XII.*

#### PATENTS.

*Phenolic aldehyde condensation products; Process for the preparation of* —. A. T. and F. E. Birkby. E.P. 153,494, 13.1.20.

A MIXTURE of 500 pts. by vol. of phenol or its homologues, 450—550 pts. by vol. of 40% formaldehyde, and 2—6% by vol. on the phenol of a mixture of 20 pts. by vol. of sulphuric acid (sp. gr. 1.84) and 80 pts. of water, is heated in a digester at 60°—80° C., with agitation for 1—2 hrs. Agitation is then discontinued, the watery layer run off, and a neutralising agent, *e.g.*, milk of lime, added in excess, with agitation. The residual water is then removed from the resinous mass by distillation under reduced pressure.—A. de W.

*Phenol resin condensates; Production of* —. F. Scudder and R. Pettigrew. E.P. 153,796, 16.12.19.

A THIN liquid phenolic condensation product, specially adaptable for the impregnation of friction blocks, stair treads, etc., is obtained by allowing sodium sulphite, 40% formaldehyde, and carbolic acid, in about the proportions of 50, 1000, and 1000 pts. by weight respectively, to react together for 3 or 4 days at ordinary atmospheric temperature with occasional stirring. The supernatant aqueous layer is removed, and the lower layer, containing 55—56% of dry resin, may be used for impregnating porous articles, which are then dried at 80°—90° C. and heated under pressure to harden the resin.  
—A. de W.

*Phenolic condensation products; Manufacture of* —. L. V. Redman, A. J. Weith, and F. P. Brock, Assrs. to Redmanol Chemical Products Co. U.S.P. 1,358,394, 9.11.20. Appl., 10.3.19.

A FUSIBLE, substantially anhydrous phenolic condensation product corresponding with a reaction between a phenolic substance and a methylene compound in proportions affording at least two phenolic groups to each methylene group, is mixed with a filler and with a methylene compound capable of reacting with the condensation product under anhydrous conditions, the methylene compound being in sufficient amount to give to the product approximately one methylene group to each phenolic group. The mixture is then subjected to a quick-moulding operation, removed from the mould, and subjected to heat treatment until a substantially infusible product is obtained.—A. de W.

*Diatomaceous earth [for use in paints]; Method of grinding* —. J. E. Schneider, Assr. to Seattle Asbesto-Mine Co. U.S.P. 1,357,480, 2.11.20. Appl., 5.5.19.

A MINERAL agent which prevents the particles of diatomaceous earth from clogging together when finely ground is added to the latter, and the mixture subjected to a very fine pulverising action. (*Cf. U.S.P. 1,317,225 of 1919; J., 1919, 916 A.*)  
—A. de W.

*Rouge; Apparatus for manufacturing* —. C. L. Waisner, Assr. to Mirror Rouge Co. U.S.P. 1,357,664, 2.11.20. Appl., 3.5.18.

THE apparatus comprises a rotary roasting chamber and a vertical support provided with a chamber having its side walls furnished with enlarged openings and its top wall with a stack leading from the chamber. The roasting chamber extends through one chamber into and across the body of the other chamber and partly into the other opening, the latter being provided with a closure.—A. de W.

*Coumarone resin; Purifying and raising the melting point of* —. Rütgerswerke A.-G. G.P. 325,575, 18.10.18.

THE resin is treated with concentrated sulphuric acid in the presence of a solvent, the solution is neutralised with compounds of alkaline earths or heavy metals, insoluble matter is separated, and the solvent is removed.—L. A. C.

*Iron compounds [pigments]; Process of manufacturing* —. E. C. R. Marks, From West Coast Kalsomine Co. E.P. 153,792, 9.12.19.

SEE U.S.P. 1,327,061 of 1920; J., 1920, 231 A.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Rubber; Action of light on* —. B. D. Porritt. *Indiarubber J.*, 1920, 60, 1159—1162.

RUBBER in balloon fabrics can be protected against the action of sunlight by the incorporation of a small proportion of litharge or of an organic dye of suitable colour. In the latter case the effect is probably one of light filtration, because a rubber solution exposed to ultraviolet light was effectively protected—as judged by its viscosity—not only when a little Sudan III. was mixed with the solution but also when the bottle containing the solution was surrounded by a benzene solution of Sudan III. The physical changes produced by the mastication of rubber are not necessarily the same as the initial effects of exposure to light and air. Masticated rubber after being stored in the dark tends to “recover” somewhat from the mechanical treatment, but oxygen appears to retard the recovery process because the surface layers of the rubber yield less viscous solutions than the interior. With solutions of lightly masticated rubber also there is a tendency to slight increase in viscosity on storage in the dark.—D. F. T.

#### PATENTS.

*Rubber; Manufacture of* —. P. Schidrowitz, W. Feldenheimer, and W. W. Plowman. E.P. 153,343, 2.6.19.

CLAY mixed with sufficient water to form a pasty mass is treated with a “deflocculating agent,” such as an aqueous solution of sodium carbonate, lime, or ammonia, and is then dried. The product forms a valuable compounding ingredient for rubber and imparts toughness and increased rate of vulcanisation.—D. F. T.

*Rubber; Processes of reclaiming* —. J. Young and W. W. Benner. E.P. 153,646, 6.8.19.

SEE U.S.P. 1,324,093 of 1919; J., 1920, 165 A.

### XV.—LEATHER; BONE; HORN; GLUE.

*Chrome tanning. II. Determination of the basicity figure of one-bath chrome liquors.* D. Barton and A. M. Hey. *J. Soc. Leather Trades' Chem.*, 1920, 4, 272—276.

CARBON dioxide functions as an acid in a chrome liquor, hence the present method (see *Leather Chem.*

Pocket Book, p. 124) of determining acid in such liquors by adding alkali to the boiling solution is inadequate, since it fails to take account of carbonic and other volatile acids. It is proposed that  $N/2$  alkali should be run into the cold diluted chrome liquor in presence of phenolphthalein and that the addition should be continued during the period of heating up to the boiling point, and then until the pink colour persists after boiling for 1 min.

—D. W.

*Gelatin gels; Theory of the tanning (hardening) process in dilute — with formaldehyde.* L. Reiner. *Kolloid Zeits.*, 1920, 27, 195–197.

Two to three per cent. solutions of formaldehyde harden 5% gelatin solutions in a few minutes, whilst a 1% solution of formaldehyde requires several days. Hardened gelatin softens at elevated temperatures, the temperature of softening being dependent on the amount of hardening and the concentration of the gelatin, but higher than the melting point of the untreated material. The softening of the hardened gelatin commences at the centre of the mass and spreads throughout the whole, and on raising the temperature sufficiently the mass becomes liquid. If the softening temperature is above  $90^{\circ}\text{C}$ ., complete liquefaction is no longer possible. The hardening takes place more readily on the surface and at the walls of the containing vessel. On cooling a melted hardened gelatin it solidifies at a temperature below the solidification point of the untreated material. The hardening process is reversible, for on heating hardened gelatin with a little water at  $100^{\circ}\text{C}$ ., formaldehyde is evolved and the gelatin regains its original physical properties. The hardening process may be stopped and reversed by the addition of small quantities of ammonia. (*Cf.* J.C.S., Jan., 1921.)—J. F. S.

*Gelatin; Swelling of — in acids.* W. R. Atkin. *J. Soc. Leather Trades' Chem.*, 1920, 4, 268–271. (*Cf.* J., 1920, 792 A.)

THE author shows that the results of Looh (*cf.* J., 1919, 331 A, 548 A; 1920, 797 A) on the effect of acid on the properties of gelatin are in close agreement with those obtained by Procter and his collaborators, and thus afford additional evidence as to the soundness of the Procter-Wilson theory of the swelling of gelatin in acids (J., 1916, 645; 1917, 92). The actual hydron concentration is the factor which determines swelling, irrespective of the nature of the acid except in the case of sulphuric acid. Other dibasic and tribasic acids behave like monobasic acids. Previous work on this subject in which hydron concentrations have not been determined is of little value. (*Cf.* J., 1918, 313 A, 314 A.)

—D. W.

*Gelatin mixtures; Electrical conductivity of — and their behaviour during the transition of gelatin.* F. Rettig. *Kolloid Zeits.*, 1920, 27, 165–172.

THE specific conductivity of gelatin-water mixtures at  $18^{\circ}\text{C}$ ., increases rapidly with increase in concentration, and approaches a maximum value. The addition of small amounts of potassium chloride, bromide, and sulphate to gelatin-water mixtures increases the conductivity to values which are greater than those of aqueous solutions of the added salts of the same concentration. When gelatin is heated to  $80^{\circ}\text{C}$ ., it is converted into  $\beta$ -gelatin, and the conductivity is irreversibly increased. The velocity of the change increases with the duration and temperature of the heating and is greater in dilute mixtures than in concentrated mixtures. (*Cf.* J. C. S., Jan., 1921.)—J. F. S.

*Gelatin; Amino-acids of —.* H. D. Dakin. *J. Biol. Chem.*, 1920, 44, 499–529.

By employing the new butyl alcohol method of extracting amino-acids (J., 1919, 196 A) considerable advances in our knowledge of the constituents of the gelatin molecule have been made. At least 91% of the molecule has now been satisfactorily accounted for, the proportions of the various amino-acids found being: glycine 25.5, alanine 8.7, leucine 7.1, serine 0.4, phenylalanine 1.4, tyrosine 0.01, proline 9.5, hydroxyproline 14.1, aspartic acid 3.4, glutamic acid 5.8, histidine 0.9, arginine 8.2, lysine 5.9, ammonia 0.4; aminobutyric acid, valine, isoleucine, and hydroxyglutamic acid were not detected. (*Cf.* J.C.S., Jan., 1921.)

—J. C. D.

#### PATENTS.

*Glue; Process of manufacturing fish —.* E. Knudsen. E.P. 153,526, 22.3.20.

FISH offal is bleached by means of a solution prepared by dissolving zinc in aqueous sulphurous acid, and is neutralised by steeping in lime water and finally cooked with water. Before bleaching, the offal may be treated with dilute hydrochloric acid, e.g., for 24 hrs. in acid of  $6^{\circ}\text{B}$ . (sp. gr. 1.043), to dissolve mineral matter, and then neutralised with lime water.—J. H. L.

*Glue; Process of manufacturing vegetable —.* V. G. Bloede. U.S.P. 1,357,310, 2.11.20. Appl., 27.11.17.

STARCH is mixed with cold water containing in solution a salt of a base which has the property of coagulating starch. The mixture is heated at or near  $100^{\circ}\text{C}$ ., until the starch is gelatinised, and the jelly formed is treated with a base capable of liberating the starch-coagulating base in order to effect a smooth and uniform coagulation of the starchy material.—J. H. L.

### XVI.—SOILS; FERTILISERS.

*Bacterial activities of the soil; Influence of moisture on the —.* J. E. Greaves and E. G. Carter. *Soil Sci.*, 1920, 10, 361–387.

THE soils used, 22 in number, ranged from a loose sand to a very stiff clay, and their content of organic matter varied considerably. In every case maximum ammonification occurred when the soil contained 60% of its water-holding capacity of water. Nitrification was at its maximum at 50–60% of water, but the results for nitrogen fixation were not so uniform, two maxima being shown by many soils, one at 50–60% and the other at 70–80% of water as compared with the water-holding capacity. Equations are given showing the relationships between the water requirements for maximum bacterial activity and the moisture equivalent, and wilting and hygroscopic coefficients as defined by Briggs (U.S. Dept. Agric., Bureau Soils Bull. 45, 1907, and Bureau Plant Ind. Bull. 230, 1912).—W. G.

*Nodule production [in legumes]; Relation of nitrates to —.* W. H. Strowd. *Soil Sci.*, 1920, 10, 343–356.

WHEN soya beans are grown in sand or soil plentifully supplied with nitrates, there is a marked accumulation of nitrates in the plant, the concentration of nitrate in the plant sap being much greater than in the soil solution. Nitrates retard, and in sufficient quantities entirely prevent, nodule formation. There is evidence that the failure of nodule production in the presence of nitrates in the soil is due, at least in part, to the effect of the high concentration of nitrate in the plant sap on

the growth and reproduction of the nodule bacteria, *Rhizobium leguminosarum*. The amount of reducing sugars in plants decreases with the increase in nitrate, but these sugars were never found to be entirely absent.—W. G.

*Nitrites and nitrates in plant tissue; Determination of —.* W. H. Strowd. Soil Sci., 1920, 10, 333—342.

THE plant tissue is extracted with cold water, and the extract divided into two portions. One portion is made alkaline with sodium hydroxide and distilled with Devarda's alloy (Cu 50, Al 45, Zn 5%). The total nitrogen present as nitrite and nitrate passes over as ammonia and is estimated. The second portion of the extract is heated in a water-bath with excess of aspartic acid for 1 hr. The nitrite is thereby destroyed and the residual nitrate is then estimated as before by distillation with Devarda's alloy.—W. G.

*Magnesia as a fertiliser; Use of —.* A. Jacob. Z. angew. Chem., 1920, 33, 292.

POTATOES were grown on soil which had been manured with nitrogenous and phosphatic fertilisers free from potassium salts, and also on the same soil similarly treated, but with the addition of potassium or magnesium salts, or both. The highest yields of potatoes and also of starch were obtained by the use of a mixture of magnesium and potassium sulphates, whilst a mixture of potassium chloride and kieserite was only slightly less effective. Sandy or peat soils were improved by the addition of magnesium sulphate.—C. A. M.

*Nitrolim; Heat changes in the formation of —.* P. Dolch. Z. Elektrochem., 1920, 26, 455—459.

THE reaction  $\text{CaC}_2 + \text{N}_2 = \text{CaCN}_2 + \text{C}$  is accompanied by a heat evolution of 58,700 Cals. per mol. of carbide. (Cf. J.C.S., Jan., 1921.)—J. F. S.

*Potassium; Volumetric estimation of —, and its application to the analysis of fertilisers.* G. Ajon. Giorn. Chim. Ind. Appl., 1920, 2, 422—426.

25 c.c. of a 2% potassium chloride or sulphate solution, equivalent to 1.26 or 1.08%  $\text{K}_2\text{O}$ , is made up to 150—200 c.c. in an Erlenmeyer flask, the liquid being then treated, slowly and with stirring, with 50 c.c. of 2N tartaric acid solution and 25 c.c. of N/2 sodium hydroxide solution. The flask is shaken for 5 mins., 25 c.c. of 96% alcohol being added gradually during the shaking. After standing for 6—8 hrs., the clear liquid is filtered through a 9 cm. barium sulphate filter paper and the precipitate washed 9 times by decantation with neutral 96% alcohol. The filter paper is removed from the funnel, used to detach any particles of precipitate from the rod, and then introduced into the precipitation vessel. A N/10 solution of sodium hydroxide in 96% alcohol is added until the contents of the vessel become alkaline to phenolphthalein. The pink colour of the liquid should not disappear within 5 mins., at the end of which time the precipitate is dissolved in aqueous N/10 sodium hydroxide solution, the excess of which is determined by titration with N/10 hydrochloric acid. Multiplication by 0.00471 of the number of c.c. of sodium hydroxide required to dissolve the bitartrate gives directly the amount of  $\text{K}_2\text{O}$  present. With commercial potassium chloride and sulphate this method gives good results.—T. H. P.

#### PATENT.

*Peat; Treatment of — and the production of a preparation for use in horticulture and agriculture.* The Molassine Co., Ltd., and H. C. S. de Whalley. E.P. 152,779, 22.7.19.

CALCIUM cyanamide, 50, is mixed with finely divided peat. 1000, and to the mixture is added calcium

carbonate, 50—200 kilos. Potassium chloride, potassium sulphate, sodium sulphate, etc., may also be added. The preparation is ready for immediate incorporation with the soil.—W. P. S.

#### XVII.—SUGARS; STARCHES; GUMS.

*Clarification of cane juice without chemical treatment.* F. W. Zerhan. La. Bull., No. 173, 1920. Int. Sugar J., 1920, 22, 643—646, 699—702.

ABSORPTION appears to be the principal factor in all the processes of clarification at present in use in sugar manufacture. Laboratory and factory experiments have proved that efficient clarification may be effected by the use of suitable adsorbing substances, such as kieselguhr, followed by a powerful decolorising carbon (as "Norit"). Using only 0.5 and 1% respectively of these substances, it was found that a sugar greatly superior in quality, and at least as high in yield, compared with that obtained in sulphitation, can be made, a molasses of higher market value than that produced by the usual plantation methods being obtained at the same time.—J. P. O.

*Settling of precipitates (aluminium hydroxide and precipitates in limed and heated cane juice).* N. Deerr. Int. Sugar J., 1920, 22, 618—624.

A SUSPENSION containing 0.1—0.5 g. of aluminium hydroxide per 100 c.c. of water when poured into a tall tube becomes resolved after about 3 min. into five zones. The first (uppermost) is quite clear, and the second is characterised by the presence of "stragglers" (particles not less than average size, which have become detached from the main body of the precipitate). The fifth (and lowest) zone is gradually being built up by the particles completing sedimentation. The fourth zone contains the main body of the precipitate, and in this zone the particles, in addition to their vertical movement, have an oscillatory or gyratory motion. The third zone contains particles that have no gyratory motion, and seem to remain unchanged until the time that the fourth zone has disappeared and the third and fifth zones meet, when after an interval the level between the clear liquid and the precipitate becomes sharp and flat, which point is termed the "critical position." That phase of settling which obtains before the critical position is reached is called the "phase of free fall," and that coming after it the "phase of restricted fall." Measurements were made of the rate of settling of suspensions of aluminium hydroxide of varying concentration in columns of liquid of different height. Cane juice which has been limed and heated gives a precipitate which subsides in a similar manner; though the variation in the quality of the juice and the quantity of lime added are factors which also affect the rate of settling. In the case of a juice having an acidity equal to 1.75 c.c. of N/1 acid per 100 c.c. (using phenolphthalein), 0.50 and 0.75 c.c. of N/1 lime suspension resulted in under-liming, while 1.25 and 1.50 c.c. gave effects typical of satisfactory defecation. Inspection of the tabulated results shows a distinct connexion between the amount of lime used and both the rate of settling and the ultimate volume occupied by the mud, the former decreasing and the latter increasing as the quantity of lime is increased. Taking the quantity of lime corresponding to 1.5 c.c. of N/1 lime suspension per 100 c.c. of juice (i.e., 0.042 g. per 100 c.c.) as representative of good practice, the conclusion is that the defecation precipitate settles at a uniform rate of 7 cm. per min.; that the critical position occurs at 0.75 of the height of the settling tank; and that the ultimate volume of the mud is about 15% of the volume of the juice.—J. P. O.

[Sugar] boiling scheme used in Java. G. E. G. von Stietz. *La. Planter*, 1920, 65, 92—93.

GOOD results are stated to be obtained by boiling three strikes, as follows: (1) Evaporator syrup mixed with second and third strike sugars, the purity being 85°—90°; (2) some of the molasses from the first strike, the purity being 70°—75°; (3) molasses from the second strike, together with the rest of the molasses from the first strike, the purity being 60°. Advantages of this method are that all the merchantable sugar originates from the first strike, giving a good and regular quality; and that in this first strike boiling is particularly easy, since it is not necessary to granulate, but only to evaporate the mixture of syrup and molasses sugar to a sufficient degree, and take in more syrup until the pan is full and until the massecuite can be transferred to the crystallisers. A waste molasses of 30°—32° is finally obtained.—J. P. O.

*Mahua flower.* Fowler and others. See XVIII.

*Botanical chemical notes.* Von Lippmann. See XX.

#### PATENTS.

*Sugar; Apparatus for the manufacture of —.* [Clarification of juices.] E. G. Aizcorve. U.S.P. 1,358,132, 9.11.20. Appl., 5.1.18.

THE apparatus comprises a heating tank, a horizontal channel, one end of which is connected with the top of the tank and the bottom of which slopes upwards towards the tank, an inlet pipe for cool juice at the other end of the channel and above the level of the tank, an outlet pipe from the tank, and a discharge opening at the end of the channel remote from the tank.—J. H. L.

*Sugar juices; Process for treating crude —.* C. Ebero. G.P. 325,220, 18.11.17.

THE juice is atomised in a current of dry air, the dried product again dissolved, the solution dialysed, and the dialysed portion and residue both again reduced to powder by drying as before. Owing to the moderate heating and rapidity of drying, both the sugar and non-sugar constituents are recovered practically unchanged.—E. H. R.

*Vegetable glue.* U.S.P. 1,357,310. See XV.

### XVIII.—FERMENTATION INDUSTRIES.

*Barley; Harvesting, storage and drying of —.* A. Cluss, W. Kluger, and V. Koudelka. Z. ges. Brauw., 1920, 43, 353—358, 361—367.

DIFFERENT portions of the same crop of Original Hannchen barley were, after harvest, treated differently in respect of time of threshing, storage, and drying, and the effects of the various treatments on the grain were investigated. It is concluded that artificial drying of the grain is a valuable means of improving its quality for agricultural and brewing purposes; in particular the germinating power of the grain is markedly increased. The barley should be stored, preferably in the ear, for about 6 weeks before being dried, to permit after-ripening. Better results were obtained by reducing the moisture content from 17.9% to 11.8% than by reducing it only to 13.8%. After storage for about 2 months (in the air) the variously treated lots of grain attained practically the same moisture content, and underwent no further perceptible change in composition.—J. H. L.

*Yeast cell; Physiology of the —.* E. Köhler. Biochem. Zeits., 1920, 111, 17—30.

YEAST previously washed with water contains an activated enzyme which produces fermentation in

sugar solution. If this enzyme is not re-activated in the process of fermentation, the fermentation comes to an end; or if the rate of re-activation is slower than the rate of utilisation of the enzyme, the rate of fermentation diminishes. The latter was found to be the case in the author's experiments. The relation of fermentation to the growth of the yeast as well as the localisation of the fermenting enzyme in the cell are discussed.

—S. S. Z.

*Enzymes; Action of — under abnormal conditions and the alleged aldehyde character of enzymes.* E. Rona. Biochem. Zeits., 1920, 109, 279—289.

THE activity of pepsin, trypsin, amylase, emulsin, invertase, and maltase was investigated in the presence of well-known carbonyl reagents in order to ascertain whether enzymes possess an aldehyde character. The results obtained show that providing that the suitable hydrogen ion concentration is insured, those enzymes generally exert their action in the presence of the reagents.—S. S. Z.

*Acetone production by Bacillus acetothyllicum; Factors that influence —.* O. F. Arzberger, W. H. Peterson, and E. B. Fred. J. Biol. Chem., 1920, 44, 465—479.

THE chief factor of importance appears to be the reaction of the medium, which for maximum acetone production should be about  $pH=5.8-6.0$ . The production of volatile acids increases when the yield of alcohol and acetone falls, and vice versa. (Cf. J.C.S., Jan., 1921.)—J. C. D.

*Cellulose; Fermentation of —.* G. J. Fowler and G. V. Joshi. J. Indian Inst. Sci., 1920, 3, 39—60.

THE conditions favourable for the most rapid formation of the largest possible quantities of methane and hydrogen by the anaerobic fermentation of cellulose were studied with the object of making economic use of this form of potential fuel occurring in the form of waste paper and vegetable debris. The ferment employed was used in the form of a "bacterial emulsion" prepared from the sludge taken from the bottom of the septic tank of a sewage works. Normal celluloses were found to be most resistant, cellulose from lignified fibres were more readily, and hemicelluloses such as banana skins or plantain skins most readily attacked. Schweitzer's reagent can be employed as a rapid test of the suitability of a cellulose material for fermentation. The efficiency and speed of the fermentation is a maximum only at a temperature near the optimum (35° C.), with a certain ratio of cellulose material and inoculant, and if the acid products of the fermentation are kept below 1% by fractional displacement of the liquor from time to time. By gradually "building up" the bacterial emulsion a daily volume of combustible gas equal to 80% of the volume of the fermenting material can be obtained with hemicelluloses. Small quantities of lead, copper, and zinc salts inhibit the fermentation. The gaseous products of the fermentation contain 80—85% of methane and have a calorific value about 1.45 times that of coal gas. The only soluble by-product that could be detected in the hemicellulose fermentation was acetic acid. It is suggested that the fermentation of cellulose waste products might probably be used as an economic source of power in such places as the western coast of India where coal is scarce.—G. F. M.

*Mahua flower; Bio-chemistry of the —.* G. J. Fowler, J. D. E. Behram, S. N. Bhate, K. H. Hassan, S. Mahdihassan, and N. N. Inuganti. J. Indian Inst. Sci., 1920, 3, 81—118.

CHEMICAL and fermentation studies were made of the sugars in the mahua flower (*Bassia longifolia* and *B. latifolia*) with a view to its utilisation as a



source of industrial alcohol. Dextrose, lævulose, maltose, sucrose, pentoses, and cellulose were identified, the total sugar being greatest when the flowers are ready to fall, at which stage it amounts in general to 60–70%. In the growing stages lævulose is present in greater amount than dextrose, but in the final stages the quantities approximate but do not become equal. Sucrose increases in amount up to the shedding of the corolla, but after this and during storage it decreases relatively to invert sugar. Numerous enzymes were detected at various stages of growth of the flower; maltase, catalase, and oxidase were present throughout. Yields of alcohol up to 90% of the theoretical were obtained by fermentation of a mash of the flowers with cultures of the natural yeast occurring in the flower, with the addition of such reagents as sulphuric acid and ammonium phosphate. By increased care in the cultivation, collection, and storage of the flowers a greater yield per tree, and a greater percentage of total sugar, and particularly of sucrose, should be possible of attainment.

—G. F. M.

*Methyl alcohol; Detection of — in liqueurs and spirits.* L. Hoton. *Ann. Falsif.*, 1920, 13, 490–491.

TWO HUNDRED AND FIFTY C.C. of the liqueur, containing at least 20% (by vol.) of alcohol, is fractionally distilled, using an efficient fractionating column, at such a rate that 10 drops of distillate per minute are collected. During the first half-minute or so the b. pt. is always low, even with pure ethyl alcohol, but rapidly reaches a value which remains constant during the first 10 mins. In the case of 25% ethyl alcohol the b. pt. during the first 10 mins. is 78° C., and is lowered by several degrees when a small quantity of methyl alcohol is present.—W. P. S.

*Botanical chemical notes.* Von Lippmann. See XX.

#### PATENTS.

*Enzyme extracts; Preparation of —.* J. Takamino and J. Takamine, jun. E.P. 152,792, 24.7.19.

THE seed spores of a suitable fungus (e.g., *Aspergillus*, *Penicillium* or *Mucor*, or *Eurotium oryzae*) are grown on a medium prepared from ground cereals; the mass is then extracted with water, the solution filtered, and the filtrate treated with sulphurous acid and heated at 45° C. in order to destroy any bacteria which are present. About 1 pt. of sulphurous acid is added to 1000–10,000 pts. of the liquid. The preparations possess marked starch liquefying and converting, proteolytic, and milk-coagulating properties.—W. P. S.

*Fermentative preparations; Process of manufacturing —.* M. Groll. E.P. 131,589, 12.8.19. Conv., 13.12.17.

SEE G.P. 308,962 of 1917; J., 1919, 510A.

#### XIXA.—FOODS.

*Milk preserved with bichromate; Composition and analysis of —.* G. Hinard. *Ann. Falsif.*, 1920, 13, 463–474.

THE addition of bichromate (0.1%) to milk retards the development of the bacteria present, but does not destroy them. For a short time the results obtained on the analysis of a bichromated milk are comparable with those yielded by the fresh milk, but decomposition soon commences and affects the constituents to varying and different extents, so that it is not possible to correct the results obtained in order to ascertain the composition of the original milk. Further, the presence of the bichromate

itself affects the determination of the total solids to an extent which varies with different milks.

—W. P. S.

*Milks; A deficiency in heat-treated —.* A. L. Daniels and R. Longhlin. *J. Biol. Chem.*, 1920, 44, 381–397.

A THOROUGH investigation of this subject shows that the well-known fact that the calcium salts are rendered insoluble on the prolonged heating of milk is responsible for the lowered nutritive value of such products. The vitamins and the food value of the proteins are apparently unimpaired.

—J. C. D.

*Bread and pastry; Leavening of —.* J. Grossfeld. *Chem.-Zeit.*, 1920, 44, 889–890.

THE characteristic and pleasant taste imparted to bread etc. by yeast may be imitated by using sour milk and a mixture of sodium bicarbonate and calcium carbonate as the leavening agent. The acidity of the milk should be about "30 degrees," equivalent to 0.67% of lactic acid; each litre of such milk used in the dough requires 14 g. of a mixture of equal weights of sodium bicarbonate and calcium carbonate.—W. P. S.

*Bread; Colloid chemistry of —.* H. Lüers and Wo. Ostwald. *Kolloid Zeits.*, 1920, 27, 34–37.

A COMPARISON of the results of the authors (J., 1920, 499 A) with those of Upson and Calvin (J., 1915, 629) and Wood and Hardy (J., 1909, 378).

—J. F. S.

*Fats; Chemical properties of nutritive — of various biological values.* S. Rosenbaum. *Biochem. Zeits.*, 1920, 109, 271–278.

No qualitative chemical differences could be established in yolk fat, cod liver oil, butter fat, fat from human milk, lard, and hazel nut oil as regards their sterol and phosphatide contents. It is therefore assumed that the biological differences of these fats are not due to differences existing in their unsaponifiable residues.—S. S. Z.

*Vitamin; Effects of water-soluble B — on nutrition.* W. G. Karr. *J. Biol. Chem.*, 1920, 44, 255–276.

A DEFICIENCY of this vitamin in the diet of the dog induces a similar type of pathological condition to that which has been observed in other species. Drying vitamin preparations at 100° C. does not appear to lower their value, but destruction occurs when they are autoclaved at 120° C. for 3 to 4 hrs.

—J. C. D.

*Vitamin; Test for anti-beri-beri — and its practical application.* C. Funk and H. E. Dubin. *J. Biol. Chem.*, 1920, 44, 487–498.

THE increased growth of yeast in the presence of extracts containing the B vitamin is utilised as the basis of a method for testing substances for the presence of the vitamin.—J. C. D.

*Vitamins; Quantitative method for determination of — in glandular and other tissues.* F. K. Swoboda. *J. Biol. Chem.*, 1920, 44, 531–551.

A MODIFICATION of the method of Williams (*J. Biol. Chem.*, 1919, 38, 465; cf. J., 1920, 259) is described and also its application to the investigation of the vitamin content of tissues. (Cf. J.C.S., Jan., 1921.)—J. C. D.

*Feeding cakes contaminated with castor oil seeds. Detection of the latter.* C. Brioux and M. Guerbet. *Ann. Falsif.*, 1920, 13, 150–160.

THE presence of castor oil seeds in feeding cakes (a dangerous contamination, since as little as 1–2% has a poisonous effect on animals) may be detected



by extracting the powdered cake several times with boiling 2% potassium hydroxide solution and examining the insoluble residue, especially the black or dark-coloured particles, under the microscope. The castor oil seed husk has a characteristic appearance. The blood-coagulating principle, ricin, present in the seeds may be separated by mixing 30 g. of the powdered cake with 150 c.c. of 0.9% sodium chloride solution and 5 c.c. of xylene, filtering the mixture after some hours, heating the filtrate at 70° C. for 1 hr., and again filtering. The filtrate is then saturated with ammonium sulphate, the precipitate formed collected, washed with 20 drops of water, dissolved in 0.9% sodium chloride solution at 37° C., and the solution filtered. The coagulating effect of the solution on defibrinated blood is then determined. Distinct coagulation occurs within 10 mins. if the cake contains as little as 2% of castor oil seeds.—W. P. S.

*Inositol-phosphoric acid of plants; Composition of* —. R. J. Anderson. *J. Biol. Chem.*, 1920, 44, 429—433.

THE composition of the salts of the organic phosphorus compound from wheat bran corresponds to the calculated composition of salts of inositol-hexaphosphoric acid,  $C_6H_8O_{14}[PO(OH)_2]_6$ . (*Cf. J.C.S.*, Jan., 1921.)—J. C. D.

*Amino-acids of gelatin.* Dakin. See XV.

#### PATENTS.

*Drying fruits, vegetables, meats, and other materials; Process of and apparatus for* —. Anhydrous Food Products Co., Assees. of O. Q. Beckworth and O. J. Hohson. E.P. 140,102. 11.3.20. Conv., 11.5.15.

SEE U.S.P. 1,228,233 of 1917; *J.*, 1917, 903.

*Furfural etc. from maize cobs.* U.S.P. 1,357,467. See XX.

### XIXb.—WATER PURIFICATION; SANITATION.

*Colloidal metal solutions.* Von Plotth. See XX.

#### PATENTS.

*Water-softener and method of preparing same.* J. E. Caps. U.S.P. 1,356,756, 26.10.20. Appl. 14.4.17. Renewed 19.1.20.

A BASE-EXCHANGING water-softening agent is prepared by heating glauconite or greensand in air to oxidise the free iron, and then removing the iron oxide from the surfaces of the glauconite particles. —W. J. W.

*Water; Sterilisation and clarification of* —. Le Manganozone Soc. Anon., Assees. of A. Tixier. E.P. 140,077, 9.3.20. Conv., 12.12.13.

SEE F.P. 474,614 of 1913; *J.*, 1915, 1112.

*Separating fats, oils, and similar matters from waste waters and the like [by settling]; Apparatus for* —. R. Poumier. E.P. 153,714, 1.9.19.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Laudanine; Constitution of* —. E. Späth. *Monatsh. Chem.*, 1920, 41, 297—304.

It has been shown that laudanine yields metahemipinic acid on oxidation, so that the two hydroxyl groups of the isoquinoline part of the molecule are methylated and the free hydroxyl group must be attached to the benzyl residue. The author finds

that ethyllaudanine, in which the phenolic hydroxyl group is protected and masked by an ethyl group, yields 4-methoxy-3-ethoxybenzoic (ethylisovanillic) acid on oxidation; similarly carbethoxylaudanine, in which the hydroxyl of laudanine is replaced by a carbethoxy group, gives carbethoxyisovanillic acid. It follows that the phenolic hydroxyl of laudanine occupies position 3 of the benzyl residue. (*Cf. J.C.S.*, Jan., 1921.)—T. H. P.

*Sinapin; Synthesis of* —. E. Späth. *Monatsh. Chem.*, 1920, 41, 271—285.

THE author has succeeded in preparing synthetically a compound of the constitution proposed by Gadamer for sinapin (*Arch. Pharm.*, 1897, 235, 93) and finds it to be identical with the latter. Various new derivatives of sinapin are described. (*Cf. J.C.S.*, Jan., 1921.)—T. H. P.

*Atractylin; Tests for the decomposition products of* —. A. Pitini. *Arch. Farm. Sperim.*, 1920, 29, 83—96. *Chem. Zentr.*, 1920, 91, IV., 614.

ATRACTYLIN,  $C_{20}H_{32}K_2S_2O_{10}$ , the poisonous glucoside of *Atractylus gummifera*, is decomposed by 10% KOH into sulphuric acid, valeric acid, carbohydrate, and an unknown substance A, which is non-poisonous to dogs and does not give Lefranc's characteristic reaction (violet colour with concentrated sulphuric acid). Consequently, in cases of poisoning with this plant, the glucoside can only be detected in the intestines in the unaltered state. The substance A, however, gives a positive reaction in presence of dextrose. Traces of A or of atractylin give with concentrated sulphuric acid containing formaldehyde a red colour which on addition of water changes to an intense blue. A sulphuric acid solution of A gives with vanilin an intense carmine-red colour. The decomposition of atractylin in the animal organism proceeds in the same way as in alkaline solution *in vitro*.—E. H. R.

*Ephedrine, pseudo-ephedrine, their optical antipodes and racemic compounds; Syntheses of* —. E. Späth and R. Göhring. *Monatsh. Chem.*, 1920, 41, 319—338.

THE authors have succeeded, by means of a series of reactions, in synthesising *d*- and *l*-ephedrine and *d*- and *l*- $\psi$ -ephedrine, which have the constitution,  $HO.CH(C_2H_5).CH(CH_3).NH.CH_3$ , and are stereoisomeric. The *l*-ephedrine thus obtained is identical with natural ephedrine, and the *d*- $\psi$ -ephedrine with natural  $\psi$ -ephedrine. It is probable, but has not yet been definitely established, that in  $\psi$ -ephedrine the hydroxyl and methylamino groups stand near to, and in ephedrine distant from, one another. (*Cf. J.C.S.*, Jan., 1921.)—T. H. P.

*Ursone and its distribution in the plant world.* A. M. Nooyen. *Pharm. Weekblad*, 1920, 57, 1128—1142.

URSONE is a general constituent of the *Ericaceæ* and is also found in some varieties of *Ilex*. It is best extracted by dilute alcoholic potash and isolated by acidification and recrystallising from hot alcohol. The molecular weight of ursone is 456, corresponding to the formula  $(C_{16}H_{14}O)_3$ . The potassium and sodium salts form white needles, readily soluble in alcohol. Ursone contains no aldehyde, ketone, or hydroxyl groups, forms no addition products, and is not affected by acid or alkaline permanganate. A nitro-product melting at 210° C. was obtained. The formula  $C_{22}H_{34}O.COOH.2H_2O$  is given. The substance and its derivatives are optically active, the potassium salt having  $[\alpha]_D^{20} = +58^\circ$ . (*Cf. J.C.S.*, Jan., 1921.)—S. I. L.

*Botanical chemical notes.* E. O. von Lippmann. *Ber.*, 1920, 53, 2069—2077.

THE formation of calcium malonate has been observed during the fermentation of a sugar solution

which was allowed to cool in a sugar refinery in consequence of cessation of work and to which a considerable quantity of clear lime water had been added. Chelidonic acid has been identified in the leaves of a species of lily termed *Gloriosa superba*, indigenous to Java. Under certain climatic conditions coumarin is found in *Melilotus arvensis* grown in Central Germany; it probably exists as a glucoside which is hydrolysed by an enzyme present in the plant and also by emulsin. A clear viscons gum found in a hole in the trunk of a mountain ash which had been struck by lightning, was found to pass rapidly into a resin (sorban) which is hydrolysed to sorbinose by the conjoint action of the fresh sap of the leaves and twigs of the mountain ash but not by the juices acting singly. Melihiose has been isolated from a clear, almost colourless syrup which, on an unusually warm day, had exuded from an incision in the stem of the mallow.—H. W.

*Aminohydroxyphenylarsenoxide; Detection of — in diaminodihydroxyarsenobenzene hydrochloride (salvarsan).* H. Cousin. *J. Pharm. Chim.*, 1920, 22, 390—392.

ONE g. of the sample is dissolved in 10 c.c. of methyl alcohol, the solution is diluted with 80 c.c. of water, 1.5 g. of calcium carbonate is added, and the mixture is shaken. The hydrochloride is decomposed and the diaminodihydroxyarsenobenzene is precipitated completely; the mixture is diluted to 100 c.c., filtered, 50 c.c. of the filtrate is diluted with 75 c.c. of water, 5 c.c. of *N/1* hydrochloric acid is added, and the aminohydroxyphenylarsenoxide is titrated with *N/20* iodine solution.—W. P. S.

*Tartaric acid from tamarinds.* J. J. Sudborough and P. N. Vridbachalam. *J. Indian Inst. Sci.*, 1920, 3, 61—80.

TAMARIND pulp contains about 12—14% of tartaric acid, part of which is in the free state and part as potassium bitartrate, and the possibility of the commercial utilisation of tamarinds in India as a source of this acid is suggested. The acid can easily be recovered by the methods used in the production of tartaric acid from wine lees, provided the pulp is first heated for 1 hr. at 160° C. in an autoclave in order to render the expressed liquor capable of being readily filtered. The yields of pure recrystallised acid obtained on a technical experimental scale amounted to about 6% on the weight of the pulp, and, in addition, about 1.7% as pure potassium bitartrate. A certain amount can also be recovered from the final mother liquors as calcium tartrate.—G. F. M.

*Lactic acid; Technique of the determination of —.* G. Riesenfeld. *Biochem. Zeits.*, 1920, 169, 247—270.

THE extraction of lactic acid with amyl alcohol according to Ohlson's method (J., 1916, 1182) yields satisfactory results when aqueous solutions are employed but not when the solutions contain protein. In such cases the author therefore recommends the precipitation of the protein with phosphotungstic acid. When the solutions are boiled a small and negligible quantity of the lactic acid is retained by the coagulum.—S. S. Z.

*Acetic anhydride; Method for estimating the strength of —.* K. Wolgast. *Svensk Kem. Tidskr.*, 1920, 32, 110. *Chem. Zentr.*, 1920, 91, IV., 597.

EXACTLY 25 c.c. of water is added to a solution of 25 c.c. of acetic anhydride in 20 c.c. of benzene, the mixture is shaken for 15 secs., allowed to settle, and the volume of the aqueous layer is measured in a burette. The acetic anhydride remains in solution in the benzene, whereas the acetic acid is extracted by the water, and thus the increase in volume  $\times 4$  gives the percentage of acetic acid in the sample.

A small correction is necessary on account of the solubility of the anhydride in water.—L. A. C.

*Urea; Transformation of ammonia into —.* C. Matignon and M. Fréjacques. *Comptes rend.*, 1920, 171, 1003—1005.

THE equilibrium pressure, *p*, in atmospheres, for ammonium carbonate heated at different temperatures in an enclosed space such that the volume occupied by the gaseous phase is as small as possible, is given by  $\log p = -1511/T + 5.6 \log T - 9.4$ .—W. G.

*Acetone and aldehyde; Determination of — in the same solution.* W. Stepp and W. Engelhardt. *Biochem. Zeits.*, 1920, 111, 8—17.

RIPPER's method (J., 1901, 288) was found to be suitable for the estimation of acetone and aldehyde in mixtures providing that at least 40 hrs. is allowed for the reaction to take place. Results obtained in the estimation of acetone and aldehyde in mixtures by the iodoform method were unsatisfactory.—S. S. Z.

*Acetone, acetaldehyde, and formaldehyde; Differential analysis of — in organic liquids.* E. Pittarelli. *Arch. Farm. Sperim.*, 1920, 29, 70—80, 81—87. *Chem. Zentr.*, 1920, 91, IV., 616—617.

A LARGE number of reactions of acetone, acetaldehyde, and formaldehyde are given. If to the solution under examination crystals of hydrazine sulphate or hydrochloride are added, then methyl orange followed by hydrochloric acid and mercuric iodide, after a few minutes a carmine-red precipitate appears if formaldehyde is present. This reaction is not given by acetone or acetaldehyde. With carbazole in alcoholic solution formaldehyde gives a green coloration in presence of concentrated sulphuric acid, not given by acetone or acetaldehyde. The aldehydes react with neutral hydrazine salts with liberation of free mineral acids, whilst acetone only gives this reaction with hydroxylamine salts. This difference can be utilised as a qualitative and quantitative test for acetone in presence of formaldehyde and acetaldehyde. Neutral colourless solutions of rosolic or brazilinic acid in sodium sulphite are coloured violet by aldehydes and acetone, through liberation of free alkali. This reaction is most sensitive with formaldehyde, least with acetone. Acetone and acetaldehyde give with hypobromite and phenols colour reactions not given by formaldehyde. They can also be identified by the isonitrile reaction or by reduction to acetylene after conversion by bromine into bromoform. Acetone is better estimated by the hydroxylamine method (titration of mineral acid liberated from a hydroxylamine salt) than by the iodoform process. A method is given for estimating free and combined acetone in urine.—E. H. R.

*Carbon; Water-soluble colloids from artificial varieties of —.* K. A. Hofmann and W. Freyer. *Ber.*, 1920, 53, 2078—2095.

COLLOIDAL preparations of an acid character have been obtained by oxidising certain kinds of carbon with potassium chlorate and hydrochloric acid. Lampblack or sawdust charcoal prepared at a low temperature was most suitable. The products were partly water-soluble, precipitated by acids; partly soluble only in dilute alkalis. The water-soluble substances were the higher oxidation products. The water-soluble product from lampblack was a monobasic acid,  $C_{11}H_8O_4$ , forming a soluble ammonium salt and an insoluble barium salt, whilst the alkali-soluble product had the composition  $C_{11}HO_4$ . The water-soluble product from charcoal had the composition  $C_{11}H_8O_4$ ; and the alkali-soluble product was a dibasic acid,  $C_{11}H_6O_4$ , forming a soluble ammonium and insoluble barium and calcium salts. The products are black to brownish-black in colour,

forming intensely coloured solutions. The colloidal particles in solution are of dimensions of the order  $2-4 \times 10^{-4}$  cm. and show well-marked Brownian movements. The products are suitable for pharmaceutical use. They powerfully adsorb iodine, some dyestuffs, and bacteria. When shaken with an alkaline solution of silver nitrate, under the influence of light, colloidal silver is adsorbed by the colloid. (Cf. J.C.S., Jan., 1921).—E. H. R.

*Colloidal metal solutions; Influence of — on lower organisms and the reason of their influence.* O. von Plotth. Biochem. Zeits., 1920, 110, 1—32.

THE behaviour of bacteria, algae, moulds, higher plants, and lower animals in metal hydrosols was investigated. A decided storage of gold was observed in the case of moulds kept in colloidal gold solutions. This entrance of the gold into the organisms was observed to be more regular and intensive in the living organisms than in the dead moulds. The storage takes place in the membrane and the gold is deposited there without entering into combination with the tissue.—S. S. Z.

*Colloidal metal solutions; Influence of — on mycelia transferred from different nutrient media.* O. von Plotth. Biochem. Zeits., 1920, 110, 33—59.

THE fixation of metallic colloids by moulds depends on the amount of protective organic colloids present. Fully protected metallic sols are not fixed at all. The storage takes place only in acid, not in alkaline media. This is explained by the fact that the fixation of the metallic colloids is a condensation process brought about by the neutralisation of the electric charge of the metallic particles by that of the particles of the hydrogel of the membrane of the moulds where the metal is fixed. The charge of the gold particles is negative; that of the particles of the hydrogels depends on the reaction of the medium. The growth of the moulds was not inhibited by gold sols, but was so by colloidal silver and copper solutions.—S. S. Z.

*Separator for use in essential oil distillation; Automatic —.* H. E. Watson. J. Indian Inst. Sci., 1920, 3, 15—18.

AN automatic separator for immiscible liquids, adapted particularly to the separation of essential oils from aqueous distillates, consists of a cylindrical vessel provided with a float kept in a vertical position by means of a stem which passes through a hole in the centre of the cover of the vessel and terminates at the bottom in a conical needle valve forming the outlet for the heavier liquid. The float is weighted to take up a position of equilibrium at the surface separating the two liquids, and the distillate is introduced horizontally into the vessel at about this point. A rise in the level of the heavier liquid lifts the float and opens the needle valve, thus allowing a discharge of the liquid until the level has again fallen to the normal position. The lighter liquid collects in the upper part of the vessel until it reaches the level of a discharge pipe, through which it continuously flows off at a rate proportional to the inflow of the distillate. A separator of this type about 8 in. in diam. and 10 in. deep is capable of dealing with about 100 lb. of distillate per hr.—G. F. M.

*Oxidation of paraffin wax.* Schaarschmidt and Thiele. See II A.

*Oxidation of hydrocarbons.* Gränacher. See II A.

*Mercuric chloride.* Kalthoff and Keijzer. See VII.

*Action of ozone on methylamines.* Strecker and Thienemann. See VII.

*Inositol-phosphoric acid.* Anderson. See XIX A.

#### PATENTS.

*Chlorhydrins and bromhydrins; Process and apparatus for the manufacture of —.* Commercial Research Co., Assees. of B. E. Eldred and B. T. Brooks. E.P. 126,311, 2.5.19. Conv., 3.5.18. (Cf. E.P. 128,578; J., 1920, 764 A.)

A GASEOUS olefine or mixture of olefines is brought into intimate contact in the lower part of a vessel or tower with a solution of hypochlorous or hypobromous acid prepared in the upper part of the tower by the action of chlorine or bromine vapour on a solution of a reagent, such as a borate, which is capable of removing the halogen acid produced according to the reversible reaction,  $Cl_2 + H_2O \rightleftharpoons HCl + HOCl$ , but which will not appreciably react with the halogen oxyacid. The solution containing the halogenhydrin, together with excess of borate, boric acid, and sodium halide, is drawn off from the base of the tower, cooled to separate the boric acid, and, after re-saturation with horax, is again circulated through the tower. The above process is repeated until a sufficient concentration of halogenhydrin has been produced in the solution, from which it is then isolated either by distillation or extraction with a solvent.—G. F. M.

*Acrolein; Process for the stabilisation of —.* C. Moureu, C. Dufraisse, P. Robin, and J. Pougnet. E.P. 141,361, 8.4.20. Conv., 8.4.19.

THE transformation of acrolein into diacryl is checked by the addition of a small proportion of phenolic or polyphenolic substances, such as pyrogallol (0.1%), catechol or quinol (0.2%), gallic acid (0.4%), or tannin (1%). Sunlight and temperatures above 30° C. should be avoided. (Cf. J., 1919, 923 A; 1920, 82 A, 173 A.)—D. F. T.

*Methane; Manufacture of —.* Farb. vorm. Meister, Lucius, und Brüning. E.P. 146,110, 21.6.20. Conv., 28.6.19.

A MIXTURE of carbon monoxide and hydrogen is passed through a series of furnaces containing a heated catalyst, the resulting water being removed and more carbon monoxide being introduced as the mixture leaves each furnace. In this way the proportion of hydrogen to carbon monoxide can be maintained in excess of 5:1, which is the limit for the avoidance of the partial decomposition of carbon monoxide into dioxide and free carbon, and yet an almost complete consumption of the hydrogen can be effected.—D. F. T.

*Ethylene chloride; Process for the production of —.* T. Goldschmidt A.-G., and O. Matter. E.P. 147,908, 9.7.20. Conv., 22.5.15.

DRY ethylene prepared by passing ethyl alcohol vapour over a contact substance, such as alumina at 400° C., is allowed to react with one-third to one-fourth of its volume of chlorine between 30° and 120° C. in the presence of a catalyst, such as chloride of iron, copper, or antimony; the temperature of the reaction chamber can be adjusted by means of a cooling device. The excess of ethylene is separated by removing the ethylene chloride in a suitable condensing plant.—D. F. T.

*Saccharin; Process for the manufacture of —.* Soc. Chim. des Usines du Rhône. E.P. 153,520, 8.3.20. Conv., 24.1.20.

By treatment with chromic acid or a dichromate, in the presence of sulphuric acid of at least 35% concentration, at a temperature dependent on this concentration, it is possible to oxidise toluene-*o*-sulphonamide to *o*-benzoic sulphimide (saccharin). It is advisable to use an excess of the amide so as to utilise the chromic acid completely.—D. F. T.

***β-Dialkylamino-ethylaminobenzoic alkyl esters; Manufacture of —.*** Soc. Chim. des Usines du Rhône. E.P. 153,827, 2.3.20. Conv., 15.11.19. Addn. to 128,554 (J., 1920, 43 A).

***β-DIALKYLAMINO-ETHYL-*p*-AMINOBENZOIC alkyl esters of the general formula, R<sub>2</sub>N.(CH<sub>2</sub>)<sub>n</sub>.NH.C<sub>6</sub>H<sub>4</sub>.CO.R', are obtained from β-chloroethyldialkylamines and *p*-aminobenzoic acid esters, by heating the two compounds together in equimolecular proportions, either with or without the use of a neutral diluent. For example, when 1 mol. of β-chloroethyldiethylamine is added to a boiling solution of 1 mol. of ethyl *p*-aminobenzoate in three times its weight of toluene, and the boiling is continued for 1–2 hrs., a solution is obtained which on cooling deposits crystals of ethyl β-diethylamino-ethyl-*p*-aminobenzoate monohydrochloride, m.p. 156° C. The compounds obtained by the interaction of β-chloroethyldiethylamine with the methyl, propyl, normal and iso-butyl, and iso-amyl esters of *p*-aminobenzoic acid are also described.—G. F. M.***

***Furfural and volatile organic acids; Manufacture of — from extracted corn [maize] cob pentosan.*** K. P. Monroe. U.S.P. 1,357,467, 2.11.20. Appl., 8.4.20.

EXTRACTED corn (maize) cob pentosan is boiled with concentrated acid, the mixture distilled, and furfural and volatile organic acids separated from the distillate by fractional distillation. The volatile organic acids are finally neutralised by alkali.

—J. S. G. T.

***Chlorination process.*** A. E. Houleban, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,358,851, 16.11.20. Appl., 26.9.17.

CHLORINE is absorbed in a hydrocarbon liquid in the absence of light, and the liquid is then heated to effect chlorination of the hydrocarbon.

—J. S. G. T.

***Fatty acid halides and phosphorus oxyhalides; Process of making —.*** F. J. Kaufmann, Assr. to C. Ritchie. U.S.P. 1,359,071, 16.11.20. Appl., 27.1.20.

A MIXTURE of the fatty acid and phosphorus trihalide is halogenated at a temperature below 50° C.

—D. F. T.

***Amylodextrin and calcium fluoride; Manufacture of a colloidal preparation of —.*** Lecinwerk E. Laves. G.P. 325,561, 12.3.19. Addn. to 323,783 (J., 1920, 802 A).

A SOLUBLE calcium salt is added to a solution of amyloidextrin and a fluoride.—L. A. C.

***Esters; Process for obtaining — from crude pyro-ligneous acid.*** Haschkowerke Eberhard & Jakob. G.P. 325,639, 10.4.18.

THE acid is neutralised and evaporated until, on cooling, sodium acetate crystallises out, the methyl alcohol present being recovered. To the further concentrated mother liquor, concentrated crude methyl or other alcohol is added with alkali bisulphate and a flux containing silicic acid, the mixture is allowed to stand and later the ester formed distilled off. The silicious flux serves to prevent superheating during distillation.—E. H. R.

****p*-Aminophenylarsinic acid; Preparation of a derivative of —.*** H. Rupe. G.P. 325,640, 25.11.17.

A PRODUCT which crystallises from alcohol in white needles, m.p. 220° C., and has a powerful action on trypanosomes and spirillæ, is prepared by heating hydroxy- or chloro-methylenecamphor with sodium *p*-aminophenylarsinate.—L. A. C.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

***Photographic plate; Photochemical investigations of the —.*** R. E. Slade and G. I. Higson. Roy. Soc. Proc., 1920, A, 98, 154–170.

SINCE it is not generally true that when light acts on a sensitive photographic film, the number of grains rendered developable is a simple function of the exposure, *I*<sub>t</sub>, it is deduced that the mechanism of the process cannot be the absorption of light in discrete quanta. If *P* is the probability that any grain is rendered developable by an exposure, *I*<sub>t</sub>, then  $\frac{d\alpha}{dt} = P(a - \alpha)$ , where *a* is the total number of grains and *α* those rendered developable. In the experimental investigation of the relationship between *P* and *I*<sub>t</sub>, plates were specially coated with a film, not more than one grain thick, of an emulsion of grains of uniform size and assumed of uniform speed. The exposed plates were developed and washed but not fixed, and the values of *a* and *α* computed by counting; from these was calculated  $\log_e \frac{a}{a - \alpha} = A$ , obtained by integrating the above equation. By varying *I*, keeping *t* constant, and plotting *A* against *I*, it was found that if *I* is great and *t* small, *A* = *k*<sub>1</sub>*I*, while if *I* is small and *t* great, *A* = *k*<sub>2</sub>*I*<sup>2</sup>. Similar relationships between *A* and *t* were obtained for exposures with constant intensity. Combining these four relationships it is deduced that  $A = k_1 I (1 - e^{-k_2 I}) (t - k_3 (1 - e^{-k_4 t}))$  which is the equation for the photochemical law of the silver halide grain.—B. V. S.

***[Colour-]sensitising properties of a new series of [cyanine] colouring matters.*** A. and L. Lumière and H. Barbier. Bull. Soc. Franç. Phot., 1920, 7, 182–184.

PARTICULARS are given of the absorption bands and sensitising properties, with spectrographs illustrating the latter, of three typical members of the series of dyes previously described by Barbier (J., 1920, 528 A). The spectrograph for Cyanine A, obtained from the ethiodides of dimethylaminoquinoline and toluquinaldine, shows maxima in the blue, yellow, and orange, and a minimum in the blue-green. Cyanine B, from the ethiodides of dimethylaminoquinoline and dimethylaminoquinaldine, shows similar maxima to Cyanine A, the action extending further into the red, and a more strongly marked minimum. Panchochrome, obtained by the condensation of dimethylaminoquinaldine with dimethylaminobenzaldehyde, shows a very uniform sensitiveness nearly up to 700 mμ, the minimum in the blue-green being very small.—B. V. S.

***Collodion; Light-sensitiveness of —.*** B. Homolka. Phot. Korr., 1920, 57, 239–241.

IN the decomposition of nitrocellulose under the action of sunlight, first demonstrated by Gladstone and Hofmann in 1852, nitric acid is formed, and this is a source of danger in some applications of celluloid or collodion. Its formation is easily shown to take place within the time of a normal photographic exposure by the aid of 9-aminophenanthrene, which is converted by very small quantities of nitric acid into the deep red 9.9' azoxyphenanthrene. Thus a collodion film containing aminophenanthrene, if exposed under a negative, yields a red positive which may be fixed in benzene in which the amino-compound but not the azoxy-compound is soluble. If a plain collodion film be exposed under a negative there is no visible result, but a latent image is formed, as is seen by treating the film with a benzene solution of 9-aminophenanthrene, drying, and then exposing the film under plain glass. A red negative, a copy of the original negative, results.—B. V. S.

[Photographic] plates; *Simultaneous development and fixing of* —. A. and L. Lumière and A. Seyewetz. Brit. J. Phot., 1920, 67, 747–748.

In preparing the combined developing and fixing solution, recommended by Otsuki and Sudzki (J., 1914, 668), containing metoquinone, sodium sulphite, sodium thiosulphate, and caustic soda, it is preferable to use a titrated caustic soda solution owing to the uncertainty of composition of the solid substance. The only other alkali which gave a satisfactory developer and is not open to this objection was tribasic sodium phosphate. Chloranol, prepared by the condensation of 2 mols. of mono-methyl-*p*-aminophenol and 1 mol. of chloroquinol, gives similar results to metoquinone.—B. V. S.

*Negative [photographic] development in bright light.* Lüppo-Cramer. Phot. Ind., 1920, 378–379.

BATHING in an amidol (diaminophenol hydrochloride) solution, 0.02 to 0.05%, for about 1 min. reduces the sensitiveness of a photographic plate from 50 to 100 times and allows subsequent development in a bright yellow light. With most developers there is hardly any effect by this previous treatment; glycin, however, gives dichroic fog, and quinol, by reason of the presence of small quantities of the amidol, becomes an energetic instead of a slow-working developer. Triaminobenzene acts similarly to diaminophenol, but the diamino-resorcinols, triaminophenol, and triaminotoluene do not produce the same effect. The addition of sulphite or bisulphite to the amidol solution reduces its desensitising effect considerably.

—B. V. S.

*Cellulose-ester films.* U.S.P. 1,357,733. See V.

## XXII.—EXPLOSIVES; MATCHES.

*Nitroso compounds; Detection of — and determination of aromatic nitro derivatives in smokeless powders.* C. Lutri. Giorn. Chim. Ind. Appl., 1920, 2, 557–559.

$\alpha$ -NAPHTHYLAMINE in 1% acetic acid solution gives an intense violet coloration with nitrous acid, nitroso-amines, and other nitroso derivatives. Certain oxidising agents, such as potassium chlorate, hydrogen peroxide, and lead peroxide also produce a violet coloration with the reagent, but these are not constituents of smokeless powders. No coloration is given by ammonium persulphate, potassium nitrate, or dinitrotoluene. For the estimation of aromatic nitro compounds 3 g. of the finely divided smokeless powder is extracted for about 6 hrs. with ether, the extract evaporated at a low temperature, and the residue heated with 50 c.c. of 20% sulphuric acid on the water bath beneath a reflux condenser, the flask being frequently shaken during 6 hrs. The liquid is filtered, the filtrate extracted with benzene, the extract evaporated at about 50° C. and the nitro derivatives weighed.—C. A. M.

*Solenite; Estimation of mineral oil in —.* R. Moretti and M. Taliani. Giorn. Chim. Ind. Appl., 1920, 2, 433–434.

FIFTY grams of the powdered explosive is boiled for 6 hrs. in a Jena flask under a reflux condenser with 400–500 c.c. of 10% sodium hydroxide. The cooled liquid is extracted several times with ether in a separating funnel, the ethereal solution being separated carefully and completely and filtered into a weighed flask; the ether is removed by distillation and the residual mineral oil dried at 105° C. until of constant weight. Good results are obtained with solenite and ballistite.—T. H. P.

*Vapour pressures; Method of measuring low — with its application to the case of trinitrotoluene.* A. W. C. Menzies. J. Amer. Chem. Soc., 1920, 42, 2218–2221.

A McLeod gauge is immersed in a constant temperature bath and connected with a bulb immersed in a second bath. The apparatus is evacuated, nitrogen is admitted up to a low pressure, and the pressure increase on heating the bulb is determined. The material is placed in the bulb, and the operation repeated, vapour being prevented from diffusing to the gauge by cooling a portion of the connecting tube. From the increase of pressure the vapour pressure of the substance may be calculated. The vapour pressures of trinitrotoluene, m.pt. 80.5° C., dried but not specially purified, were calculated to be as follows in mm. mercury at 0° C.:—(80° C., 0.042 mm.); (81°, 0.044); 82°, 0.046; 83°, 0.048; 84°, 0.051; 85°, 0.053; 86°, 0.056; 87°, 0.059; 88°, 0.062; 89°, 0.064; 90°, 0.067; 91°, 0.070; 92°, 0.074; 93°, 0.078; 94°, 0.081; 95°, 0.085; 96°, 0.089; 97°, 0.093; 98°, 0.098; 99°, 0.102; 100°, 0.106; 101°, 0.110; 102° C., 0.116 mm. These values were obtained graphically from observed pressures, 0.046 mm. at 81.6° C., 0.067 mm. at 90.1° C., and 0.120 mm. at 102.6° C.—J. R. P.

*Nitro products; Decomposition of —.* S. Brown. Chem. Trade J., 1920, 67, 673.

In a laboratory investigation into the cause of an explosion on a nitration plant, it was found that the presence of lubricating oil in a nitration mixture accelerates the rate of decomposition by acid of the nitro product, e.g., dinitrotoluene.—L. A. C.

### PATENTS.

*Gelatinous nitroglycerin explosives proof against fire-damp; Manufacture of —.* Dynamit A.-G. vorm. A. Nobel und Co., and P. Naoum. E.P. 140,746, 12.12.19. Conv., 22.3.19.

GELATINOUS nitroglycerin explosives of a plastic nature, and safe for use in presence of fire-damp, are prepared, without the use of amylaceous or similar materials, by adding concentrated solutions of hygroscopic salts, advantageously mixed with a little glycerin, to the explosive constituents. A suitable composition consists of:—Nitroglycerin, 30%; 50% calcium nitrate solution, 3%; glycerin, 1%; ammonium nitrate, 30%; sodium chloride, 35%; woodmeal, 1%. This mixture gives a lead block expansion of 220 cm., and has a charge limite of 650 g. It remains unchanged during storage.

—W. J. W.

*Amatol; Method and apparatus for removing — from shells or like explosive bodies for the purpose of recovering ammonium nitrate.* C. F. Beakbane and J. W. Arnot. E.P. 153,123, 14.8.19.

A WATER tank provided with a steam coil has an outlet pipe in communication with a centrifugal pump and a supply pipe terminating in nozzles over which are placed the shells to be emptied. Below these nozzles is placed a trough which drains into a settling tank with an overflow outlet into the water tank. A branch pipe from the centrifugal pump passes into another settling tank from which liquid can be conducted into an evaporating tank and crystallising vessels. Water at 86° C. and under a pressure of 25–55 lb. per sq. in. is pumped from the water tank through the nozzles into the shells, from which the mixture of ammonium nitrate solution and trinitrotoluene flows round the nozzles through the trough into the settling tank where sludge is deposited. The solution is returned to the water tank and circulated through the shells until it reaches a density of 50° Tw. (sp. gr. 1.250), after which it is pumped through the branch pipe into the second settling tank where the trinitrotoluene is eventually precipitated. The ammonium

nitrate solution is then run off into the evaporating tank, concentrated to 72° Tw. (sp. gr. 1.380), and then passed into the crystallising pans.—W. J. W.

**Ammonium perchlorate explosives.** R. M. Cook and E. W. Harris, Assrs. to Atlas Powder Co. U.S.P. 1,357,764, 2.11.20. Appl., 2.9.20.

THE composition of the explosives is as follows:—Ammonium perchlorate, of which at least 50% passes a 60-mesh sieve, and coated with 0.1 to 1.5% of vegetable or mineral waxes or oils, 15–65%; manganese dioxide, 3–15%; sodium nitrate, coated with 0.1–1.5% of vegetable or mineral waxes or oils, 5–50%; liquid aromatic nitro-compounds, 0.5–5%; trinitrotoluene, 15–30%; sulphur, 0–7%; chalk, 0–3%. The proportions of the ingredients are selected so as to give an oxygen balance of the explosive between –4% and +6%. —W. J. W.

**Nitrocellulose; Process of stabilising — and product thereof.** C. L. Reese, Assr. to The Arlington Co. U.S.P. 1,358,653, 9.11.20. Appl., 8.12.16.

NITROCELLULOSE or soluble nitrocellulose may be stabilised by the addition of 0.1–2% of dicyanodiamide, with or without a solvent, or camphor, or both.—W. J. W.

**Nitration acid from the manufacture of nitrocellulose or the like; Recovery of —.** S. Hamburger. G.P. 299,680, 12.5.16.

AFTER nitration the bulk of the acid is allowed to flow away and the material is washed, without centrifuging, with sulphuric acid of the same strength as the waste acid, so that on mixing the two lots of acid there is no evolution of heat. —L. A. C.

### XXIII.—ANALYSIS.

**Ultrafiltration apparatus.** L. S. Villegas. Contr. Estud. Ciencias [La Plata], Ser. mat. fis., 1919, 2, 415–418. Chem. Zentr., 1920, 91, IV., 614–615.

A GLASS cylinder, 9 cm. high and 7 cm. diam., is closed at the top and bottom with bronze discs and rubber rings to make a tight joint. In the upper disc are two openings for a manometer and pressure pump respectively. On the bottom, between the rubber ring and bronze disc, is a thin metal disc of platinum, aluminium, or nickel, perforated with a number of holes 1 mm. in diam., to support the ultra-filter. The latter is prepared by completely immersing 12.5 g. of guncotton in absolute alcohol, and adding ether to bring the volume up to 500 c.c. Part of this solution is poured on to a glass plate, and when it has coagulated it is soaked in water, and the thin layer removed from the plate. Such filters are permeable to colloids, the permeability depending on the acidity; 0.5% of acetic acid greatly improves the permeability. Collodion filters were also successfully used, their permeability depending not only on their acidity, but on the time elapsing after the addition of acid. The apparatus is specially useful for colloidal solutions of metals, and has also been used for solutions of hæmoglobin, albumin, and milk.—E. H. R.

**Thoulet's solution; Substitute for —.** A. Thiel and L. Stoll. Ber., 1920, 53, 2003.

FOR the determination of the specific gravity of organic substances by the suspension method, an aqueous solution of lead perchlorate can be used in place of the more expensive Thoulet's solution (mercury-potassium iodide) or Rohrbach's solution (mercury-barium iodide). The perchlorate solution is prepared by saturating commercial perchloric acid with commercial lead carbonate and evaporating to the saturation point. The solution satur-

ated at 15° C. is a mobile liquid, sp. gr. 2.6, and contains 78% of lead perchlorate.—E. H. R.

**Cresol red; Salt error of —.** R. C. Wells. J. Amer. Chem. Soc., 1920, 42, 2160–2167.

IN the determination of the hydrogen ion concentration of salt water by a colorimetric method with cresol red as indicator, it is necessary to make a correction for the effect of the salt on the colour of the indicator. This correction has been determined with buffer solutions of borax and boric acid without and with sodium chloride, and a table of corrections for use with the indicator is given. —J. R. P.

**Alkali metals; Determination of — as sulphate.** N. Schoorl and I. M. Kolthoff. Chem. Weekblad, 1920, 17, 425–427.

VAN DUIN (*ibid.*, 283) has stated that errors arise in the ignition of alkali pyrosulphate to sulphate, and proposes the evaporation of a solution of ammonium carbonate with the pyrosulphate, and ignition of the residue. It is shown that this introduces even greater errors, and that if a fragment of ammonium carbonate is added to the crucible in which the residue, after careful evaporation of the acid, is maintained at the maximum temperature without melting, volatilisation occurs quietly and evenly, and very accurate results may be obtained. (Cf. J.C.S., Jan., 1921.)—S. I. L.

**Barium, strontium, and calcium; Detection of — by the chromate method.** I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 1080–1084.

THE three elements can be satisfactorily detected by the procedure given; 1 mg. of either in presence of 100 mg. of each of the others per l. can be identified. Barium is precipitated by addition of bichromate to a solution containing acetic acid and sodium acetate, strontium from the filtrate by addition of ammonia and alcohol, and calcium by the oxalate method. (Cf. J.C.S., Jan., 1921.) —S. I. L.

**Lead; Determination of — as chromate.** I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 934–941.

THE lead is precipitated as chromate, a small quantity of aluminium chloride being added to improve the separation. For small quantities, the precipitate is filtered off, dissolved, after washing, in hydrochloric acid, excess of potassium iodide added, and the liberated iodine estimated by thiosulphate. For larger quantities, the excess of chromate may be estimated in the same manner, since large precipitates of lead chromate are not easily handled. The method is quick and accurate, and may be applied to lead sulphate, which is dissolved in sodium acetate and acetic acid. Where the lead is combined with a strong acid, conductivity determinations may be made, since the chromic acid liberated by the action of potassium bichromate functions as a strong monobasic acid. In this method, the liberated chromic acid is titrated with soda, the conductivity diminishing until all the acid has been converted into bichromate, and then slowly increasing. (Cf. J.C.S., Jan., 1921.) —S. I. L.

**Electro-analytical practice. Determination of mercury.** W. Böttger. Z. Elektrochem., 1920, 26, 445–452.

MERCURY is quantitatively deposited from solutions of mercurous nitrate, in the presence of nitric acid, when a constant EMF of 1.4–1.5 volts is employed. From mercuric nitrate solutions to which 1 c.c. of nitric acid (sp. gr. 1.4) has been added, it is deposited quantitatively when the EMF is so regulated that the current remains at 1 ampère. To ensure this the electrolysis is started with an EMF of 1.8 volts, which rises to

3.0 volts in about 4 mins. and finally reaches 3.2—3.3 volts, at which value the electrolysis is completed. Pure alcohol and pure ether may be used for drying the deposit if the electrode is exposed to the air for only a short time (10—15 mins.). When a silvered net electrode is used the loss in the above processes is very constant and amounts to 0.4 mg. Separation from a cyanide solution is accompanied by a loss of 0.6 mg. The accuracy of the cyanide method is somewhat doubtful when a large amount of potassium cyanide is present and when a large current is used.—J. F. S.

See also pages (A) 3, *Calorific power of gas* (Evans). 7, *Potassium iodide and mercuric chloride* (Kolthoff), *Mercuric chloride* (Kolthoff and Keijzer). 11, *Portland cement* (Ferrari). 14, *Babbitt metal* (Boehmer and Others), *Metallurgical products* (Compagno). 19, *Chrome tanning liquors* (Burton and Hey). 21, *Nitrites and nitrates in plant tissue* (Strowd), *Potassium* (Ajon). 23, *Methyl alcohol in spirits* (Hoton), *Anti-beri-beri-vitamin* (Funk and Dubin), *Vitamin* (Swoboda). *Feeding cakes* (Brioux and Guerbet). 24, *Attractylin* (Pitini). 25, *Amino-hydroxyphenylarsenoxide in salvarsan* (Cousin), *Lactic acid* (Riesefeld), *Acetic anhydride* (Wol-gast), *Acetone and aldehyde* (Stapp and Engelhardt), *Acetone, acetaldehyde, and formaldehyde* (Pittarelli). 28, *Smokeless powders* (Lutri). *Mineral oil in solenite* (Moretti and Taliani), *Vapour pressures* (Menzies).

### Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

#### I.—GENERAL; PLANT; MACHINERY.

##### APPLICATIONS.

Acheson. Deflocculating solid materials, and agents therefor. 35,064. Dec. 13. (U.S., 22.1.20.)  
 Braden Copper Co. Filtering. 36,103. Dec. 23. (U.S., 2.1.20.)  
 Brutzkus. Effecting chemical reactions in compressors. 35,975. Dec. 22. (U.S., 22.12.19.)  
 Burdick. Spraying and drying apparatus. 36,046. Dec. 23.  
 Cleworth, Wheal, and Co., and Leask. Purification of air and gas. 35,646. Dec. 18.  
 Drey, and Williams and Co. Catalysts and catalytic reactions. 36,189—36,191. Dec. 24.  
 Hoyle. Centrifugal driers. 35,548. Dec. 17.  
 Hussey. Grinding mills. 35,997. Dec. 22.  
 Mazza. Separation of gaseous mixtures. 35,862. Dec. 21.  
 Plauson and Vielle. Filter presses. 36,169. Dec. 24. (Ger., 5.7.18.)  
 Plauson and Vielle. Manufacture of dispersoids. 36,171. Dec. 24. (Ger., 8.6.19.)  
 Rigby. Evaporation of liquids containing solids in solution or suspension. 36,183. Dec. 24.  
 Slatineanu. Catalytically obtaining reactions between a gas and another substance. 35,461. Dec. 16. (Switz., 5.11.20.)  
 Soc. d'Utilisation des Combustibles Pulvérisés. Rotary drying apparatus. 35,921. Dec. 22. (Fr., 6.11.20.)  
 Sturgeon. Centrifugal separators. 35,033. Dec. 13.

##### COMPLETE SPECIFICATIONS ACCEPTED.

21,348 (1919). Russell. Crushing-machines. (154,975.) Dec. 22.  
 21,095 (1919). Allsop and Sibson. Drying-machines. (154,986.) Dec. 22.  
 21,881 (1919). Zimmermann and Weyel. Drying-apparatus. (154,998.) Dec. 22.

22,530 (1919). Sokal (Naaml. Vennoots. Verschure & Co.'s Scheepswerfen Machinefabr.). Mills for grinding, crushing, or pulverising. (132,789.) Dec. 30.

25,284 (1919). Carborundum Co. See VIII.  
 7335 (1920). Lowden. Drying or partially eliminating moisture from materials. (155,493.) Dec. 30.

9797 (1920). Metcalfe. Tunnel-kilns or furnaces. (155,164.) Dec. 22.

14,785 (1920). Lewis and Green. Apparatus for separating a volatile liquid from solution in a relatively non-volatile liquid. (155,511.) Dec. 30.

#### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

##### APPLICATIONS.

Adler. 35,172 See XX.  
 Barrs. Low-temperature distillation and apparatus therefor. 35,748, 35,753, 36,129. Dec. 20 and 23.  
 Barrs. Manufacture of briquettes. 36,130. Dec. 23.  
 Bates. Liquid fuel. 36,168. Dec. 24. (U.S., 12.4.20.)  
 Blake. Alcohol fuels. 35,447—8. Dec. 16.  
 Broadbridge and Stenning. Concentration of coal and filtration of the concentrates. 35,139. Dec. 14.  
 Brownlee and Ganahl. Treating hydrocarbon oils. 36,126. Dec. 23.  
 Carpenter. Plant for manufacture of gas. 35,334. Dec. 15.  
 Clarkson. Burning powdered fuel. 35,521. Dec. 17.  
 Cleworth, Wheal, and Co., and Leask. 35,646. See I.  
 Coke and Gas Ovens, Ltd., and Kimbell. Regenerative coke ovens. 35,165. Dec. 14.  
 Cuthbert. Manufacture of decarbonising agents for internal-combustion engines etc. 35,224. Dec. 14.  
 Emerson. Conversion of hydrocarbon oils. 35,959. Dec. 22.  
 Griffiths. Machines for manufacture of composition fuel. 35,572. Dec. 18.  
 Haersolte van Haerst. Coke ovens. 36,007. Dec. 22. (Ger., 27.12.19.)  
 Hughes. Distillation of oil etc. from shale etc. 35,874. Dec. 22.  
 Krüger and Kubrak. Process of combustion. 35,848. Dec. 21.  
 Nicol. Producing gaseous fuel. 36,200. Dec. 24.  
 Parker. Gas-producers and carbonisers. 35,744. Dec. 20.  
 Rigby. Treatment of peat etc. 36,032. Dec. 23.  
 Rollason. Production of compound fuel oils. 36,157. Dec. 24.  
 Smith and Tulloch. Gas-producers. 35,220. Dec. 14.  
 Smith. Suction-gas generators. 35,266. Dec. 15.  
 Soc. Gén. de Fours à Coke. Regenerative coke ovens. 35,909. Dec. 22. (Belg., 13.3.20.)  
 Southcombe and Wells. Lubricating oils. 35,500. Dec. 17.  
 Trent. Treatment of carbonaceous materials. 35,332. Dec. 15. (U.S., 25.2.20.)  
 COMPLETE SPECIFICATIONS ACCEPTED.  
 15,803 (1919). Jackson (Koppers Co.). Coking-retort ovens. (153,316.) Dec. 30.  
 22,054 (1919). Goskar and Thomas. Treatment of peat, brown coal, etc. (155,012.) Dec. 22.  
 22,785 (1919). Rincker. Water-gas producers. (139,451.) Dec. 30.  
 25,276 (1919). Young. Low-temperature distillation of coal. (134,529.) Dec. 30.  
 12,030 (1920). Schmidt. Treatment of paraffin



wax to produce oxygenated compounds. (142,507.) Dec. 30.

15,192 (1920). Traube. Extraction of ethylene from gaseous mixtures. (147,543.) Dec. 22.

20,584 (1920). Sudfeldt u. Co. See XX.

### III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Scheibler. Manufacture of sulphur preparations of the thiophene series. 35,523. Dec. 17. (Ger., 24.11.15.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,568 (1919). Moore. Reduction of substituted nitro compound, nitroso and azo compounds. (155,319.) Dec. 30.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

British Dyestuffs Corp., Green, and Saunders. Manufacture of soluble acid colouring matters and intermediate compounds. 35,647. Dec. 18.

British Dyestuffs Corp., and Fyfe. Manufacture of 1-chloro-2-aminoanthraquinone. 35,853. Dec. 21.

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,568 (1919). Moore. See III.

24,870 (1919). Brotherton and Co., and Merri-man. Manufacture of disazo colouring matters. (155,410.) Dec. 30.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

British Cellulose and Chem. Manuf. Co., and Richardson. Treatment of products made with cellulose derivatives. 35,629, 35,630. Dec. 18.

British Research Assoc. for Woollen and Worsted Industries, and Shorter. Treatment of wool etc. 35,245. Dec. 15.

Claessen. Manufacture of waterproofing-material. 36,002. Dec. 22. (Ger., 1.4.19.)

Claessen. Manufacture of elastic material from nitrocellulose. 36,003. Dec. 22. (Ger., 3.9.19.)

Köln-Rottweil A.-G. Manufacture of cellulose material. 36,001. Dec. 22. (Ger., 30.12.19.)

Turner. Treatment of fibrous substances. 35,076. Dec. 13.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### APPLICATIONS.

Clarenbach. Textile fabric-treating machines. 35,944. Dec. 22.

Clavel. Dyeing cellulose acetate. 35,196. Dec. 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1738 (1920). Minton. See XIV.

7844 (1920). Vallaëys. Dyeing-vats. (140,764.) Dec. 22.

### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

#### APPLICATIONS.

Collier. 36,255. See X.

Goedicke. 35,633. See XI.

Nitrogen Corp. Process of synthesising ammonia. 35,720. Dec. 20. (U.S., 20.12.19.)

Norsk Hydro-Elektrisk Kvaestofaktieselskab. Production of concentrated nitrous gases. 35,222. Dec. 14. (Norway, 5.5.20.)

Rollason. Production of ammonia. 35,876. Dec. 22.

Soc. l'Air Liquide. Direct synthesis of ammonia. 35,354. Dec. 15. (Fr., 15.12.19.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

15,149 (1919). Rollason. Production of ammonia. (155,313.) Dec. 30.

22,079 (1919). Gans. Production of iron-free ammonium alum. (132,510.) Dec. 22.

22,825 (1919). Arrowsmith. See XIII.

3206 (1920). Kereszty and Wolf. Production of solid basic magnesium hypochlorite. (142,081.) Dec. 30.

### VIII.—GLASS; CERAMICS.

#### APPLICATIONS.

Banks. Non-steaming glass. 35,603. Dec. 18.

Highfield, Laurie, and Osmosis Co. Mining or concentration of clay. 35,796. Dec. 21.

#### COMPLETE SPECIFICATIONS ACCEPTED.

19,288 (1919). Tucker and Reeves. Method of getting viscous glass-charges. (131,586.) Dec. 30.

25,234 (1919). Carborundum Co. (Linbarger). Manufacture of grinding wheels from ceramic or refractory materials. (155,076.) Dec. 22.

### IX.—BUILDING MATERIALS.

#### APPLICATIONS.

Dalhoff and Lunn. Manufacture of material for use in making light concrete. 35,993. Dec. 22.

Graeffe and Haase. Preparing building-material from peat. 35,155. Dec. 14.

Jackson (Bitoslag Paving Co.). Paving mixtures. 36,100. Dec. 23.

Polla. Manufacture of building materials with ligneous fragments. 35,075. Dec. 13. (Switz., 13.12.17.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

24,757 (1919). McDowall. Fireproof flooring or the like. (155,072.) Dec. 22.

27,648 (1919). Baylor. Slow-setting fat and waterproof cement, and method of producing same. (155,431.) Dec. 30.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

#### APPLICATIONS.

Collier. Extraction of metallic compounds from blast-furnace etc. slags. 36,255. Dec. 24.

Collins. Separation and recovery of metals. 35,632. Dec. 18.

Counas. Electric treatment of ores. 35,992. Dec. 22. (Fr., 29.8.19.)

Goskar and Hitch. Manufacture of compositions for case-hardening iron and steel. 36,193. Dec. 24.

Hall, and Rolls-Royce, Ltd. Aluminium-bronze alloys. 35,509 and 36,177. Dec. 17 and 24.

Houmøller. Briquetting cast-iron chips. 35,814. Dec. 21. (Denmark, 19.8.20.)

Hurley. Process of galvanising or zinc-dipping. 36,028. Dec. 23.

Loosli and North. Production of zirconium. 35,326. Dec. 15. (Ger., 14.11.19.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

19,316 (1919). Madsen. Electro-deposited metals. (142,432.) Dec. 22.

22,370 (1919). Wood, and Minerals Separation, Ltd. Apparatus for the froth flotation process. (155,349.) Dec. 30.

22,959 (1919). Fletcher. Manufacture of puddled iron. (155,042.) Dec. 22.

27,785 (1919). Akt. Ferrolegeringar. Production of chromium or its alloys. (135,187.) Dec. 22.

3279 (1920). Rader, and Akt. Zink. Electric furnaces for producing zinc. (139,160.) Dec. 30.

15,584 (1920). Krupp A.-G. Grusonwerk. Extracting metals from compounds, ores, furnace dust, etc. (144,728.) Dec. 30.

16,559 (1920). McKenna. Purification of ferro-tungsten. (146,108.) Dec. 30.

17,690 (1920). Ayestaran é Irazusta. Conglomeration of pulverulent mineral residues. (155,522.) Dec. 30.



## XI.—ELECTRO-CHEMISTRY.

## APPLICATIONS.

British Thomson-Houston Co. (General Electric Co.). Manufacture of resistance material. 35,620. Dec. 18.

Counas. 35,992. *See* X.  
Ges. f. Teerverwertung. Manufacture of electrodes of large cross-section. 35,319. Dec. 15. (Ger., 27.11.19.)

Goedicke. Ozon-generating apparatus. 35,633. Dec. 18.

Leitner. Electric accumulators. 35,436. Dec. 16.  
Plauson and Vielle. Electrodes and electrolysis. 36,170. Dec. 24. (Ger., 6.7.18.)

## COMPLETE SPECIFICATIONS ACCEPTED.

16,907 (1919). Whalley and others. *See* XIII.  
24,718 (1919) and 6654 (1920). Oldham and Oldham. Galvanic batteries. (155,071.) Dec. 22.

32,791 (1919). Levin. Electrolytic cells. (155,118.) Dec. 22.

32,794 (1919). Levin. Electrolytic apparatus. (155,457.) Dec. 30.

1840 (1920). Comp. Franç. des Métaux. Electric induction furnaces. (138,604.) Dec. 30.

3217 (1920). Tagliaferri. Electric furnaces. (155,476.) Dec. 30.

3279 (1920). Rader, and Akt. Zink. *See* X.

## XII.—FATS; OILS; WAXES.

## APPLICATIONS.

Bigum. Rotary-cooling apparatus for fatty substances, emulsions, etc. 35,818. Dec. 21. (Denmark, 22.12.19.)

Böhme A.-G. Production of fat-dissolving substances. 35,723. Dec. 20. (Ger., 19.12.19.)

Chambers and Hammond. Treatment of wool fat etc. 35,591. Dec. 18.

Hcy. Removing suspended matter from oils and solutions of oils. 35,382. Dec. 16.

Schou. 35,553. *See* XIX.

Starrels. Production of fatty acids of high melting point. 36,081. Dec. 23. (U.S., 1.3.16.)

## COMPLETE SPECIFICATIONS ACCEPTED.

22,080 (1919). Gans. Manufacture of saponaceous cleansing composition. (132,511.) Dec. 22.

22,159 (1919). Vakil. Refining oils and fats. (155,020.) Dec. 22.

3814 (1920). Clayton and Nodder. Cooling apparatus for use in the manufacture of edible fats. (155,477.) Dec. 30.

14,358 (1920). Schoonderwaldt. Preparation of substitutes for linseed oil. (155,508.) Dec. 30.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

## COMPLETE SPECIFICATIONS ACCEPTED.

16,907 (1919). Whalley, and Micanite and Insulators Co. Recovery of varnish and other ingredients from waste micanite etc. (155,318.) Dec. 30.

22,825 (1919). Arrowsmith. Manufacture of acid pots for use in making white lead. (155,373.) Dec. 30.

23,964 (1919). Schou, and Emulsion Aktieselskabet. Manufacture of painting, priming, and like compositions. (155,398.) Dec. 30.

27,254 (1919). British Thomson-Houston Co. (General Electric Co.). Japans. (155,427.) Dec. 30.

14,358 (1920). Schoonderwaldt. *See* XII.

20,584 (1920). Sudfeldt u. Co. *See* XX.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

## APPLICATIONS.

Baines. Rubber mixings. 35,474. Dec. 17.  
Roa, Ltd., and Wickham. Apparatus for treating latex. 36,128. Dec. 23.

## COMPLETE SPECIFICATIONS ACCEPTED.

1738 (1920). Minton. Rubber-proofed fabrics. (155,469.) Dec. 30.

4240 (1920). Hunter Dry Kiln Co. Treatment of rubber. (138,915.) Dec. 23.

## XV.—LEATHER; BONE; HORN; GLUE.

## APPLICATIONS.

Earp. Tanning. 35,099. *See* IX.

Harley. Composition for dressing leather etc. 35,159. Dec. 14.

## COMPLETE SPECIFICATION ACCEPTED.

19,058 (1919). Marris, and Walker and Sons. Treatment of refuse from tanneries etc. (154,961.) Dec. 22.

## XVIII.—FERMENTATION INDUSTRIES.

## APPLICATIONS.

Gille, Nathan, and Nathan-Institut A.-G. Cooling and aerating beer wort and separating sludge. 36,263. Dec. 24.

Verein der Spiritus-Fabrikanten. Production of yeast. 35,282—3, 35,285—7, 35,289, 35,290, 35,292—4. Dec. 15. (Ger., 18.3, 24.2, 31.3, 19.3, 15.3, 7.5, 23.12, 12, 15, and 23.4.15.)

Verein der Spiritus-Fabrikanten. Treatment of yeast. 35,284. Dec. 15. (Ger., 26.8.15.)

Verein der Spiritus-Fabrikanten. Production of compressed yeast. 35,288. Dec. 15. (Ger., 19.8.15.)

Verein der Spiritus-Fabrikanten. Treatment of froth of fermenting or boiling liquids. 35,291. Dec. 15. (Ger., 19.8.16.)

## XIX.—FOODS; WATER PURIFICATION; SANITATION.

## APPLICATIONS.

Andrews and Paul. Treatment of flour etc. 36,237. Dec. 24.

Quaker Oats Co. Alimentary paste. 35,963. Dec. 22. (U.S., 9.7.20.)

Schou. Manufacture of margarine and edible fats. 35,553. Dec. 17.

## COMPLETE SPECIFICATIONS ACCEPTED.

27,285 (1919). Borrowman. Water-softening. (155,092.) Dec. 22.

3814 (1920). Clayton and Nodder. *See* XII.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

## APPLICATIONS.

Adler. Production of adsorptive charcoal for medicinal purposes. 35,172. Dec. 14. (Czechoslov., 5.12.19.)

Fabr. de Prod. Chim. de Thann et de Mulhouse. Manufacture of borneol. 36,260. Dec. 24. (Fr., 23.5.20.)

Spitz. Manufacture of therapeutic preparations of calcium iodide. 36,035. Dec. 23. (Ger., 10.3.16.)

Stockholms Superfosfat-Fabr. Akt. Manufacture of acetaldehyde from acetylene. 35,970. Dec. 22. (Sweden, 16.12.19.)

## COMPLETE SPECIFICATION ACCEPTED.

20,584 (1920). Sudfeldt u. Co. Obtaining the salts of sulpho-acids and alkylsulphuric acids from acid resins. (148,579.) Dec. 30.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## APPLICATIONS.

Janzer. Photographic printing. 35,619. Dec. 18.

Kent. Photography. 35,988. Dec. 22.

## XXIII.—ANALYSIS.

## APPLICATION.

Irving and Thorpe. Calorimeters. 35,716. Dec. 20.

**I.—GENERAL; PLANT; MACHINERY.****PATENTS.**

*Rectifiers for rectifying mixtures of liquefied gases; Process and apparatus for regulating the operations in —.* E. Barbet et Fils et Cie. E.P. 129,648, 9.7.19. Conv., 9.7.18.

THE apparatus is provided with devices (pressure, temperature, and speed regulators, meters, etc.) for maintaining regularity of operation, these devices being controlled automatically by the pressure differences existing in the various parts.

—W. E. F. P.

*Refrigerating apparatus.* C. Delaguer. E.P. 138,933, 13.2.20.

THE apparatus is of the type in which cold is produced by the vaporisation of a liquid which has previously been removed (as a gas) from a solvent liquid by boiling or heating and afterwards condensed. A straight pipe of comparatively small diameter leads from the boiler-absorber to the downward sloping portion of the return pipe from the condenser-refrigerator to the boiler-absorber, which latter pipe is continued downwards into the boiler-absorber, ending in a perforated nozzle near the side of the vessel below the liquid level. The condenser-evaporator may be combined in one vessel which takes the form of a jacket surrounding a chamber for the material to be chilled. The jacket should be narrow for the greater part of its height, but very wide for a short distance at the top, to give both a large surface for absorbing heat from the material and a large surface for evaporation of liquid; or the condenser and evaporator may be two separate coils. In this case by means of suitable cocks and valves the main part of the apparatus may be removed, leaving only the evaporating coil attached to the refrigeration chamber.—B. M. V.

*Furnaces; Rocking —.* J. Lambot. E.P. 141,054, 31.3.20. Conv., 3.4.19.

A ROCKING furnace is combined with a recuperator in one structure so that the whole can be tilted, thus avoiding joints between the recuperator and the furnace.—B. M. V.

*Kiln.* J. Elcock. U.S.P. 1,329,830, 3.2.20. Appl., 8.9.19.

A KILN, circular in plan, is provided with chimneys in the walls spaced regularly around the periphery, and connected by approximately radial passages or slots in the floor with various points in a pair of parallel openings extending across the floor of the kiln near to, but on each side of its centre. Coal is coked in chambers arranged outside the kiln walls, and the coke pushed back into "flash walls" or boxes inside the kiln where it is burnt. The hot gas from these walls circulates downwards in the kiln and passes out through the radial slots in the floor.—W. F. F.

*Furnace.* C. L. W. Trinks, Assr. to Tate-Jones and Co. U.S.P. 1,360,058, 23.11.20. Appl., 2.2.20.

A FURNACE comprises a heating chamber provided with a metallic hearth having heat-resisting material immediately underneath. A combustion chamber is situated below the heating chamber.

—J. S. G. T.

*Ovens; Device for reversing gaseous currents in [regenerative] —.* E. Lecocq, Assr. to Soc. Gén. de Fours à Coke Systèmes Lecocq. U.S.P. 1,361,595, 7.12.20. Appl., 18.7.17.

THE gaseous currents in a regenerative oven may be reversed by arranging the gas and air flues in pairs one above the other, so that when a double

sliding damper is in one position the gas and air flow through one regenerator, and when the damper is reversed they flow through a second regenerator.

—A. B. S.

*Regenerator.* W. Gast. U.S.P. 1,361,709, 7.12.20. Appl., 12.12.19.

THE regenerator consists of a number of chambers in each of which a construction of chequer work is arranged, one of the chambers communicating with the inlet and another with the outlet of the apparatus. One of the chequer constructions is provided with gas-distributing channels situated close to the gas inlet and relatively larger in cross-sectional area than the respective chequer passages in the same chamber.—W. E. F. P.

*Volatile solvents; Methods of recovering —.* H. Schmidt. E.P. 141,739, 15.4.20. Conv., 7.8.15.

SOLVENTS evaporated during the drying of substances or articles are absorbed by an agent flowing through the drying chamber, and are subsequently recovered. The liquid absorbent may, for example, be allowed to flow along rods, threads, wires, textile fabrics, or the like arranged within the drying chamber.—W. E. F. P.

*Volatile solvents; Removal of — from materials treated therewith.* H. Bollmann. E.P. 154,044, 21.10.19.

IN a process of removing volatile solvents by conveying the material under treatment along a rising path against a counter current of steam, the material is subjected to alternate heating and cooling to enable the steam to penetrate better. Apparatus for this purpose may consist of an enclosed and inclined worm conveyor, the casing of which is formed of alternate hot and cold jackets.

—B. M. V.

*Evaporators for liquids; Heat interchange devices such as —.* E. Wirth-Frey, Assee. of A.-G. Kummeler und Matter. E.P. 142,077, 21.1.20. Conv., 23.4.19.

AN evaporator has vertical heating channels formed by annular, hollow, heating bodies arranged concentrically one within the other; the channels are provided with scrapers which extend from above and operate in a resilient manner on the vertical walls. The scrapers are of clamp shape, removable, and mounted on a horizontal beam which is rotated about a vertical axis.—W. E. F. P.

*Concentration, evaporation, and similar operations [e.g., for sulphite-cellulose waste lyes].* E. C. R. Marks. From Aktiebolaget Indunstare. E.P. 154,355, 3.9.19.

CONCENTRATION is effected in two or more stages by repeated circulation of the liquid as a thin film over a surface heated by the evolved vapour after compression of the latter to raise it to the necessary temperature, the solution being subjected to similar temperatures and pressures in the different stages, for the purpose of decreasing the total surface required for a given duty. The process is designed for use especially with liquids (such as sulphite-cellulose waste lye), the concentration of which increases the viscosity without affecting the boiling point appreciably.—W. E. F. P.

*Evaporating process.* J. G. Stead. E.P. 154,762, 14.10.19.

A PAIR of drying rolls, mounted to rotate side by side, has a trough-like duct arranged directly beneath the line of contact. Each roll has a casing on its under side, and the inside edges of the two casings are joined to the adjacent edges of the central trough. Between each roll and its casing

is an air space which gradually diminishes from the outside (or intake) edge towards the central duct; the latter is provided with a suction pipe for the purpose of drawing air from the atmosphere through these spaces, and with a drain pipe at its lowest part. The liquid to be evaporated is fed to the V-shaped space between the upper sides of the rolls.—W. E. F. P.

[Centrifugal] separators for granular and pulverulent materials, matter in suspension and the like. H. Hooke. E.P. 154,038, 9.10.19.

THE basket of a centrifugal apparatus consists of two truncated cones with a common base and with sides forming acute angles with the base so that material shall not stick to the walls. Openings in the basket may be left at or just below the largest circumference for the discharge of concentrate, or the latter may be left in for removal later, the tailing in each case being discharged over the top edge of the basket. An annular feed member rotating in the same direction as the basket hut at a different speed, delivers the material near the bottom of the basket and is continued upwards again as a flange parallel to the lower conical wall of the basket, and may be provided with stirring vanes. Alternatively, a diaphragm may be placed at the common base of the two cones, the feed being just above this, and holes left in the diaphragm near the circumference, so that only the concentrates will pass downwards.—B. M. V.

Centrifugal separator. C. P. Tolman, Assr. to National Lead Co. U.S.P. 1,358,897, 16.11.20. Appl., 2.4.17.

THE separating chamber comprises two relatively separable sections between which the heavier material escapes through a peripheral annular outlet. Openings are provided in one of the sections for the escape of lighter material, and means are provided for rotating one of the sections relatively to the other.—J. H. L.

Separating liquids and solids; Method of and apparatus for —. J. Avrutik. U.S.P. 1,360,708, 30.11.20. Appl., 2.7.19.

A CONTINUOUS stream of the mixture is fed into and through a series of receptacles mounted in cascade fashion on the same vertical shaft, and is progressively subjected in successive compartments to a greater centrifugal liquid separating force, which is employed to feed the mixture from one receptacle to the next.—J. S. G. T.

Air saturating towers [for saturating air-blast with steam]. H. Nielsen and F. D. Marshall. E.P. 154,310, 21.8.19.

A CLOSED, cylindrical tower provided with an inlet and an outlet for air at the lower and upper end, respectively, has an internal tube arranged concentrically, the annular space being packed with filling material. The inner tube is closed at the bottom, hut has a lateral inlet pipe for water a short distance above, and is divided into sections by partitions. Each of the latter has an opening from which a tube depends into the next section below, and a valve discharge-pipe extends laterally from the top of each section through the annular space and the wall of the outer tower. The inner tube is open at the top, upon which a water-distributing device is disposed. The apparatus is designed to prevent clogging of the packing material by suspended matter in the water employed, part of the suspended matter being separated at the bottom of the inner tube and drawn off through a sludge cock, and the remainder trapped beneath the successive partitions and discharged through the lateral tubes.—W. E. F. P.

Cylinders for drying food; paper and such like. J. McIntyre, and J. Milne and Son, Ltd. E.P. 154,508, 12.4.20.

THE ends (or plates covering the ends) of the cylinders are coated with glass or with vitreous or stove enamel, in order to facilitate cleaning.—W. E. F. P.

Drying apparatus. H. Wettig, Assr. to J. A. Topf und Soehne. U.S.P. 1,359,301, 16.11.20. Appl., 26.8.20.

MATERIAL is dried in a vertical shaft having reticulated walls for the passage of air. The shaft is divided into a number of sections, and the air is directed horizontally backwards and forwards through successive sections from the bottom to the top. Means are provided for passing either hot or cold air through the material.—W. F. F.

Drying machine. T. Allsop and W. W. Sibson, Assrs. to The Philadelphia Drying Machinery Co. U.S.P. 1,360,705, 30.11.20. Appl., 4.9.19.

A DRYING machine comprises a large chamber divided lengthwise into a series of zones through which the articles to be dried are successively passed. Means are provided for maintaining different temperatures and degrees of humidity in the various zones, and for circulating air within the chamber, the circulation in the first zone being more sluggish than in the others.—J. S. G. T.

Crystal production. R. W. Moore, Assr. to General Electric Co. U.S.P. 1,347,350, 20.7.20. Appl., 26.2.18.

TO produce large and perfect crystals from solutions the saturated solution, at about 32°–35° C., is heated about 7°–8° C. above its saturation temperature, filtered, and placed in a vessel contained in a water bath surrounded by an electric heating coil. Small fragments of crystals are then suspended in the solution, which is maintained at about 0.5° C. above the saturation point. The setting of the thermostat by which the temperature is regulated is changed each day to produce a drop in temperature of about 0.1° C. per day, while evaporation is prevented. The decrease in temperature may be increased to a maximum of 0.6 C. per day after the crystals have grown to large size.—W. F. F.

Grinder and pulveriser; Compound —. S. H. Herbst, jun. U.S.P. 1,358,739, 16.11.20. Appl., 9.3.20.

A GRINDING machine is provided with fixed inner and outer grinding rings, with a revolving series of grinding wheels between the rings. Alternate wheels are pressed by yielding means against the outer and inner rings, respectively, and each ring is provided with a sifting screen.—W. F. F.

Ball tube-mill; Combination —. J. E. Kennedy. U.S.P. 1,360,648, 30.11.20. Appl., 12.10.17.

A COMBINED ball and tube mill comprises a rotary tube with a sectional lining and a partition screen formed of a series of sectors, each sector having a relatively broad peripheral rim extending laterally on both sides of a perforated medial web. The lateral edges of the rims are in direct contact with the lining of the tube. The sectors of the partition are held rigidly at the centre and by the lining, and are thus prevented from moving longitudinally within the tube.—H. S. H.

Separator. J. T. Costello. U.S.P. 1,359,578, 23.11.20. Appl., 22.5.18.

A ROTARY drum having a porous periphery is partly immersed in a tank containing the mixture to be separated, and a vacuum is created within the drum whereby the liquid is drawn into it and the solid

deposited on its periphery. Means are provided for removing the solid deposit and for successively introducing compressed air into certain parts of the drum to clean the filtering medium.—W. F. F.

*Slime-pulp thickener.* C. Allen. U.S.P. 1,360,703, 30.11.20. Appl., 10.10.18.

A BAG made of flexible material is contained within a settling tank provided with an orifice for the discharge of settled material, and communicates with a float chamber above. The float is actuated by the change in density of the medium surrounding the bag, and controls the size of the discharge orifice. Means are provided for registering the variation in the density of the pulp surrounding the bag. The buoyancy of the float may be adjusted, and the float may be connected with a discharge orifice so that liquid of a predetermined density may be discharged.—J. S. G. T.

*Suspensions and colloids; Process for purifying — by dialysis.* W. Müller. G.P. 323,960, 21.3.19.

THE liquid is evaporated considerably during dialysis by means of a vacuum or by heating it, *e.g.*, by blowing a current of hot air over the surface. Crystalloids are thus rapidly separated from the colloid.—A. R. P.

*Alkali-resisting articles and apparatus.* R. Rosenberg. G.P. 326,032, 19.2.19.

MELTING pots, tubes, evaporators, and the like to withstand the action of alkalis are made from practically chemically pure, carbon-free iron, *e.g.*, electrolytic iron, which has been melted in a vacuum or purified by any special process. Alloys of pure iron with nickel, tungsten, or the like are also suitable for making alkali-resisting apparatus. Sheets of pure nickel lost 0.5% of electrolytic iron, 2.75%, and of Siemens-Martin iron, 26.5%, when fused with potassium hydroxide for 100 hrs.—A. R. P.

*Roller-mill.* F. E. Marcy. U.S.P. 1,361,601, 7.12.20. Appl., 25.1.19. Renewed 13.10.20.

SEE E.P. 136,950 of 1919; J., 1920, 391 A.

*Gas and liquid contact device.* C. J. Goodwin. U.S.P. 1,360,928, 30.11.20. Appl., 19.1.18.

SEE E.P. 110,260 of 1917; J., 1917, 1228.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal; Calculation of the efficiency of complete gasification from the analysis of —.* H. Strache and A. Gross. Mitt. Inst. Kohlenvergasung, 1920, 2, 4—5, 45—46, 50—53, 59—61, 68—71. (Cf. J., 1920, 621 A.)

THE efficiency of the various methods of gasification were calculated for different varieties of coal, taking as typical cases the production of semi-water gas, Mond gas, and compound gas ("double gas" or "tri-gas" processes). The efficiency calculated on tar-free coal varies (for the coals cited), for semi-water gas between 66.0 and 78.7%, for Mond gas between 47.2 and 52.1%, for compound gas ("double gas") without pre-drying and with excess steam between 74.0 and 83.7%, and for compound gas with pre-drying and without excess steam between 54.8 and 69.8%. In the semi-water gas process, by the use of warm water for moistening the air, an efficiency of 65—80% can be attained, according to the ash and water content of the coal; whilst in the Mond gas process the efficiency varies from 45 to 55%. The compound gas ("double gas") process, without pre-drying, is only practicable if the ratio of the water content of the coal to the ash-free coke is less than 2.60. If this ratio is smaller than

0.5, 10—20% excess steam can be used. The total efficiency lies between 80 and 85%. The compound gas process with pre-drying is only practicable if the ratio of water to ash-free coke is more than 2.60, or if the yield of ammonia makes it worth while. The efficiency of this process is 70—75%. The efficiency is not, however, the only criterion of the value of a process. Other factors may be of more importance, *e.g.*, the calorific value of the gas (especially where it is necessary to attain high temperatures), or the ammonia yield, which is high in the Mond process, although the efficiency is low. The quality of the tar again varies greatly according to the process adopted. The compound-gas process would be used where high-flame temperatures are necessary, where the gases are to be conducted to long distances, or where the yield and quality of the tar are of great economic importance.—A. G.

*Cholesterol; Contact decomposition of —. Theory of petroleum formation.* W. Steinkopf, H. Winternitz, W. Roederer, and A. Wolynski. J. prakt. Chem., 1919, 100, 65—85.

THE view that the optical activity of petroleum originates from cholesterol and its derivatives accounts for most of the facts, with the exception that whilst the decomposition products hitherto prepared from cholesterol are all dextro-rotatory, certain fractions of petroleum exhibit laevo-rotation. It is now found, however, that if cholesterol be heated alone at 150°—200° C., or in presence of kieselguhr at 150° C., the laevo-rotation gradually diminishes to a minimum and then increases; in presence of quartz at 200° C. the diminution is continuous, and eventually dextro-rotatory products are formed. It is suggested that under the conditions prevalent in nature cholesterol furnishes first a laevo- and ultimately a dextro-rotatory cholesterylene, which subsequently give products, some richer, some poorer in hydrogen.—J. K.

*Petroleum; Specific heat of — at different temperatures.* F. W. Bushong and L. L. Knight. J. Ind. Eng. Chem., 1920, 12, 1197—1200.

THE specific heats of petroleum products at different temperatures are recorded graphically and also in tabular form. For solid paraffin the value varies from 0.12 at 210° C. to 0.47 at 0° C.; for liquid paraffin the value increases from 0.5700 at 52° C. to 0.6307 at 100° C. For petroleum distillates the values 0.4703 at 21.1°C. and 0.5420 at 68° C. are given.—W. P. S.

*Petroleum, tar, etc.; New method of fractional distillation of —.* H. Offermann. Chem.-Zeit., 1920, 44, 773—774.

THE author modifies the usual method of obtaining the heavy fractions of petroleum and similar materials, namely, distillation with superheated steam, by using wet steam at 100° C., and claims to achieve higher yields of more viscous oils and smaller amounts of pitch. The explanations he suggests are that cool steam (plus entrained water) represents a greater quantity of water than superheated steam per unit volume; and that the evaporation of entrained water droplets within the oil increases the efficiency of contact of steam with oil.—A. E. D.

*Gasoline left in residual gas from compression plants; Absorption as applied to the recovery of —.* W. P. Dykema and R. O. Neal. U.S. Bureau of Mines, Tech. Paper 232, 1920. 40 pp.

THE absorption method of recovering gasoline is more efficient than compression, and is applicable either to rich gas (3 to 6 galls. of gasoline for 1000 cb. ft.) or to the residual lean gases from compression plants. Using naphtha as absorbent a plant to handle 500,000 cb. ft. per day can be in-

stalled for less than \$1000. Details are given of satisfactory units, baffling devices, packing, rates of flow, and the recovery of gasoline from the absorbent by distillation. Portable testing apparatus for determining the gasoline content of natural gas is also described.—A. E. D.

*Paraffin wax; Analysis of* — K. Bube. Petroleum, 1920, 16, 5–8. Chem.-Zentr., 1920, 91, IV., 350.

THE specific gravity at 50° C. of lignite paraffin wax increases nearly linearly with the addition of dark paraffin oil, and it has been found that the difference between the sp. gr. at 50° C. and 0.784 in units of the third decimal place gives the percentage content of dark paraffin oil in hard paraffin wax (m.p. about 50°) is used. With soft paraffin wax and intermediate qualities the figures 0.781 and 0.782, respectively, should be substituted. The results are accurate to 1–2% provided that the content of oil is under 41%. The method is useful for controlling the sweating process.—A. E. D.

*Viscosities of liquids at high pressures; Apparatus for determination of absolute — and results obtained with it for certain lubricating oils.* J. H. Hyde. Report of Lubricants and Lubrication Inquiry Committee. (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 80–84.

IN the method described a closed system of two horizontal tubes (the upper one capillary in dimensions) and two vertical tubes is used. The lower half of the system contains mercury and the upper half the liquid under investigation. Any desired pressure and temperature may be maintained in the apparatus. One end of the system rests on a horizontal knife edge and the other is carried by a spiral spring. On the mercury being displaced by a given amount, flow will take place round the circuit owing to difference of head, and if the spring be so designed that its rate of extension is equal to the change of head of the mercury, flow of the liquid under test will take place through the capillary under constant pressure difference and at a velocity calculable from the rate of extension of the spring. All the data needed for the determination of absolute viscosity are thereby obtained. Illustrations are given showing the construction of the viscosimeter, the general arrangement of the viscosimeter and heating bath, and a pressure intensifier for use with the apparatus. All the oils tested showed rapid rise of viscosity with pressure (see following table in which  $\eta$  = viscosity and  $\rho$  = density):—

	Pressure, tons per sq. in.	Value of $\eta/\rho$ C.G.S. units.	Temp., ° C.
Castor oil	1.44	227.5	40.2
"	5.40	264.5	40.0
"	7.39	1164.0	40.5
"	(atmospheric)	1.94	—
Mobiloil B.B. (mineral)	1.44	227.5	40.0
"	4.51	710.0	39.8
"	6.47	1019.0	40.0
"	(atmospheric)	1.58	40.0
Bayonne (mineral)	1	157.5	39.9
"	4	430.0	40.0
"	8	1260.0	40.0
"	(atmospheric)	0.49	40.0

—A. E. D.

*Lubricating oils; Compressibility of — under high pressures, and the application of the results to the tests in the high-pressure viscosimeter to obtain the values of the viscosities of the oil.* J. H. Hyde. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 86–90.

THE expression  $\eta/\rho$  in the previous report (cf. *supra*) was used in the absence of data relative to the densities of the material under high pressures. To obtain such data a steel cylinder filled with the liquid under test and fitted with a sleeved plunger was used, this being immersed in the liquid con-

tained in a strong tube and the pressure in the tube raised to the desired amount. When the pressure was released the motion of the plunger was determined by its position relative to the sleeve, which was held by springs in the new position created by the applied pressure. The compressibilities of the oils and of water were determined and were found not to differ materially from one another:—

Pressure, tons per sq. in.	Densities at 40° C.			
	Castor oil.	Sperm oil.	F.F.F. cylinder.	Mobiloil B.B.
0	0.9415	0.896	0.877	0.899
4	0.9685	0.896	0.904	0.920
8	0.9905	0.9195	0.926	0.949
10	1.001	—	—	—

—A. E. D.

*Viscosity of liquids at high pressure; Absolute —. Results of tests on rape, sperm and F.F.F. cylinder oils.* J. H. Hyde. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 85.

RAPE and sperm oil do not materially differ from castor oil in their behaviour; similarly the mineral oil resembled the mobiloil and Bayonne lubricating oil previously tested (cf. *supra*).—A. E. D.

*Viscosity of oil at high pressure; Testing the —.* C. A. Parsons. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 98–99.

THE shape and position of oil grooves in the bush seem to have a great effect on the friction. In turbine bearings large oil grooves are provided, and the heat is carried away by pumping a large volume of oil through the bearings, the oil being cooled and then returned to the bearings.—A. E. D.

*Viscosity of oils under any pressure; Apparatus for examination of —.* C. V. Boys. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 100–101.

A DRIVEN tubular shaft carries at its end within a chamber in a strong steel block a cylindrical shell, the exterior surface of which is the inner surface of the oil film. The outer oil film surface is formed by the inner surface of a sleeve surrounding the shell. The sleeve is carried by a gimbal ring held by a slender tubular shaft in line with the driven shaft and free to turn in the cover of the steel block, and a tubular extension of the same. The tubular extension is partly cut away to carry a block, which is provided with a pointer and counterweight.

—A. E. D.

*Viscosimeter; Cup and ball —.* T. C. Thomsen. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 104–110.

THE most accurate results are obtained when the ball and cup are both immersed in oil heated to a desired temperature. The cup is pressed on the ball, the instrument suspended in the oil, and the time taken for the ball to drop is ascertained. The rougher method, in which a few drops of oil are inserted between the cup and ball, the two parts pressed together and then suspended and the time interval of contact noted, is less accurate. Particular care must be taken with regard to temperature regulation, and if proper care is taken results comparable with those given by Redwood's viscosimeter are obtainable.—A. E. D.

*Oil emulsions; Memorandum on Patterson's suggested method of preventing the separation of — when mixed with salt water.* Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 115.

"ACME" cutting compound (emulsion of mineral

oil, soap, and water) was diluted with water and with salt solution of various concentrations. Salt up to 0.2% did not cause appreciable separation of the emulsion. When salt was present in amounts sufficient to cause separation, the addition of gelatin, as suggested by Patterson, did not prevent the separation.—A. E. D.

*Graphite; Effect of — on lubricants. Tests on the Lanchester worm gear testing machine.* J. H. Hyde. Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 75–79.

FLAKE graphite gave marked improvement in efficiency when added to some oils and little change with others. Where improvement was shown it was in excess of that obtained with "oildag." Broadly speaking, "oildag" and flake graphite had a beneficial effect under the conditions of the tests. Even when they did not materially raise the efficiency, however, they appeared to have the effect of raising the temperature at which a serious fall in efficiency occurred.—A. E. D.

*"Oildag" in aero-engines; Trials of — by the Air Ministry.* Report of Lubricants and Lubrication Inquiry Committee (Dept. of Sci. and Ind. Res. Advisory Council), 1920, 117–124.

THE results of the tests indicate that "oildag" helps towards maintaining good running conditions, full power output over an extended run, and reduction in mechanical losses, but the saving is only a small fraction of the total power output, and is probably more than counterbalanced by the accompanying disadvantages.

#### PATENTS.

*Coal and the like; Apparatus for washing —.* R. A. Henry. E.P. 153,790, 4.12.19.

WATER charged with coal or ore is supplied by a centrifugal pump to one end of a trough having an aperture in its bottom through which another stream of water flows upwards. The heavier particles fall through this opening into a receptacle from which they are removed by a bucket conveyor. The lighter particles pass onward through the trough to an outlet at the other end, while the unclassified material passes through other apertures in the bottom, and thence back to the pump in a closed circuit for further classification.—W. F. F.

*Coal or other mineral washing apparatus.* A. France. E.P. 153,983, 27.8.19.

COAL is washed in a number of superposed troughs, each provided with separators, and the schist etc. is discharged from the lowest trough to a reservoir. The solid material in the reservoir, consisting of heavy material from the upper troughs and light material from the separators of the lowest trough, is lifted by an elevator to the uppermost trough for further washing.—W. F. F.

*Coal and like material; Jig for separating — from foreign matter.* C. V. King. U.S.P. 1,360,116, 23.11.20. Appl., 17.4.19.

A SEPARATING chamber with an inclined perforated floor is formed as a compartment in a tank, and water is forced upwards through the floor by a piston reciprocating in another compartment in the tank. A settling chamber is provided at the forward end of the inclined floor, and has two inclined faces, which direct the material to an outlet between them. The settling and separating chambers are separated by a vertical partition, the lower end of which is spaced from the floor so as to leave a passage between the two chambers. Water and lighter materials overflow above the settling chamber, and another portion of the separated

material collects behind a vertically adjustable plate at the rear of the separating chamber.

—W. F. F.

*Fuel; Composition —.* D. B. Macdonald, and The Densified Peat Fuel and Products, Ltd. E.P. 154,289, 18.8.19.

A MIXTURE of moist peat (1 pt.), reduced to a pulp with or without the addition of hot or cold water, and coal dust (1 pt.) is worked in a pug-mill, formed into blocks, and dried. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 2237 of 1873, 654 of 1875, 1806 of 1889, 17,154 of 1890, 16,027 of 1902, and 122,214.)—L. A. C.

*Artificial fuel; Manufacture of —.* J. W. Leadbeater. E.P. 154,319, 23.8.19.

PEAT (168 lb.) containing 50–60% of moisture, from the black, lower peat strata, is disintegrated and mixed with about 8 lb. of pitch, 4 lb. of ground slaked or unslaked lime, and 270 lb. of fine coal. The product is ready for use without compression into briquettes.—L. A. C.

*Coke-oven heating wall and the like.* A. Roberts, Assr. to American Coke and Chemical Co. U.S.P. 1,358,787, 16.11.20. Appl., 27.1.19.

A COKE-OVEN heating wall is built up of courses of blocks, each having a recess extending over two of its adjoining faces, the recesses in adjacent blocks being contiguous. Each block is provided in the corner opposite to the recess with a flat portion for jointing to a corresponding flat portion of a brick in the next course. The jointing portions have their edges bevelled, and their jointing faces are alternately at the top and bottom of the course, while all face in the same direction along the length of the wall.—W. F. F.

*Coke and the like; Method relating to the quenching of hot —.* R. and J. Dempster, Ltd., and G. F. H. Beard. E.P. 153,838, 31.3.20.

HOT coke is carried by a conveyor which moves just below the surface of the water in a tank, so that the coke is quenched by a comparatively small amount of water or by the steam generated from it. The forward edges of the conveyor plates may be bent downwards to disturb the surface of the water and wash it against the coke. Means may be provided for varying the level of the quenching liquid or the height of parts of the conveyor.—W. F. F.

*Carbonisation or destructive distillation [of finely subdivided fuel]; Method of and apparatus for —.* Underfeed Stoker Co., Ltd., and S. McEwen. E.P. 154,253, 18.6.19.

POWDERED coal is carbonised to a finely subdivided coke as follows. The powdered fuel is conveyed into the bottom of an upwardly expanding chamber by a current of the gas produced in carbonisation mixed with the hot products of the combustion of part of this gas in a separate furnace with a minimum excess of oxygen. The upward blast produces a relative movement between the gas and the particles and allows the larger particles the longest time for carbonisation. The carbonised dust is separated in a "cyclone" and the gases scrubbed for oil.—C. I.

*Carbonisation of finely subdivided fuel.* Underfeed Stoker Co., Ltd., and S. McEwen. E.P. 154,458, 18.6.19.

POWDERED coal is carbonised in such a manner that it retains its powdered form, e.g., by allowing it to fall freely in a vertical retort, and is then showered into a current of the gas produced in the carbonising retort and conveyed by it into a combustion chamber.—C. I.

*Suction gas producer.* H. Bloomfield and F. J. Morgan. E.P. 154,436, 14.11.19.

A GAS producer is surrounded by a double jacket. The exhaust gases from the gas engine circulate through the outer jacket and serve along with the heat of the producer to raise steam in the inner jacket. A supplementary gas-fired boiler may be provided in the form of an annular chamber around the lower part of the producer.—C. I.

*Gas; Apparatus for the manufacture of* — M. C. Whitaker and W. F. Rittman, Assrs. to Synthetic Hydro-Carbon Co. U.S.P. 1,348,766, 3.8.20. Appl., 28.2.16.

LIQUID substances which are to be brought into reaction, e.g., mineral oils, are admitted to the upper end of a vertical cylinder containing a quantity of small pieces of refractory material supported on a perforated plate. The main part of the cylinder is unobstructed and is surrounded by an electric heating coil, the whole cylinder being heat-insulated. The upper part of the cylinder is heated sufficiently to gasify the liquid instantaneously, and the products pass out at the bottom to a condenser and thence to a collecting vessel. Means are provided for controlling the pressure and temperature within the reaction chamber.—W. F. F.

*Producer gas; Process for making or treating* — W. B. Chapman and D. Mason, Assrs. to Chapman Engineering Co. U.S.P. 1,361,137, 7.12.20. Appl., 16.3.15.

THE fuel from which the gas is produced is led into a heated gasifying chamber, and the resulting gases, while hot, are subjected to the action of an ionising medium in a separate chamber.—A. J. H.

*Gas-washing apparatus.* H. Neilsen and F. D. Marshall. E.P. 154,309, 21.8.19.

A SCRUBBING tower is provided on both sides with shelves inclined inwards and extending nearly to the opposite side of the tower. There are two vertical revolving shafts each carrying a spraying basket for every compartment in the tower. Curtains of perforated plate, metal strips, chains, or strings of beads are hung between the spraying baskets. The shelves are provided with ridges to guide the liquid collecting on them through the openings in the shelves for the shafts and into the baskets underneath.—C. I.

*Gas; Purification of* — [from naphthalene]. C. E. Holt and J. S. Burgoyne. E.P. 154,742, 20.9.19.

GAS is passed through a helical screen of wire gauze of gradually increasing fineness. The meshes are sufficiently coarse not to become clogged with tar, and films of oil form in them and absorb naphthalene. The deposited matter drops off and is removed, and the degree of purification is regulated by the temperature.—C. I.

*Gas; Purification of* — [from sulphur compounds]. H. L. Doherty. U.S.P. 1,360,734, 30.11.20. Appl., 22.5.17.

NATURAL or other gas is purified from acid gases without loss of pressure as follows. It is scrubbed under pressure with a stream of ammonia solution, the resulting liquor heated, the ammonia removed from the gas given off, and the ammonia and water returned to the scrubber.—C. I.

*Mineral oil; Process of purifying used* — The De Laval Separator Co., Asses., of C. H. Hapgood. E.P. 142,089, 6.4.20. Conv., 23.4.19.

OLEIC acid (3 lb.) is added to dirty lubricating oil (100 gals.), from which gasoline, if present, has been previously removed by treatment with steam or hot air, and the mixture is agitated at 180° F. (about 80° C.). A solution of 1½ lb. of soda ash and

1½ lb. of sodium chloride or sulphate in 9 galls. of water is added, and the oil is separated from the aqueous solution, which contains the suspended carbon and dirt from the oil, by centrifugal means.—L. A. C.

*[Motor] fuel; Non-freezing* — E. C. R. Marks. From U.S. Industrial Alcohol Co. E.P. 153,925, 7.5.19.

A FUEL for internal combustion engines, which is non-freezing at -60° F. (-51° C.), consists, according to one example, of ethyl alcohol 40 pts., gasoline 28 pts., benzol 17 pts., ether 7.5-10 pts., and toluol 8 pts. In other examples kerosene and methyl-ethyl ketone are used as ingredients.—W. F. F.

*Fuel; Non-freezing* — E. C. R. Marks. From U.S. Industrial Alcohol Co. E.P. 154,867, 7.5.19. (Cf. 153,925, *supra*.)

AN internal combustion engine fuel which does not freeze above about -60° F. (-51° C.) consists of 12 pts. by vol. of benzene, 30 pts. of gasoline, and 40 pts. of absolute alcohol.—L. A. C.

*Hydrocarbon oil; Treatment of* — N. V. S. Kuibhs. E.P. 154,464, 20.12.19.

HYDROCARBON oil is decomposed, with formation of products of lower boiling point, by heating with an alkali metal, e.g., sodium, or an alloy of the same, e.g., sodium-lead alloy, either in a still fitted with a fractionating column, or in an autoclave, or by passing the oil vapour through the molten metal.—L. A. C.

*Hydrocarbon oils; Apparatus for treating* — M. J. Trumble, Assr. to Shell Co., of California. U.S.P. 1,349,794, 17.8.20. Appl., 19.7.16.

Oil is sprayed into a separator through an opening through which hot furnace gases are also introduced in the same direction.—A. E. D.

(A, c) *Hydrocarbons; Apparatus for cracking* — (a) *Cracking hydrocarbons.* J. W. Coast, jun., Assr. to Process Co. U.S.P. (a) 1,349,815, (b) 1,349,816 and (c) 1,349,817, 17.8.20. Appl., 27.9.17.

(A) A FIRE-HEATED cracking still is provided with a circulating pump and filter both inside the still. The pump is operated by means outside the still and withdraws oil from the bottom of the still, forces it through the filter to remove coke-forming matter, and returns the filtered oil to the main body of oil. (A) A pressure still operating at more than 3 atm. pressure has an arcuate bottom provided with scrapers whereby the coke is continuously moved from the middle to opposite sides of the still and accumulated at high levels away from the hottest portion of the bottom of the still. (c) A scraping device for a still of the type described in (a), consists of a series of chains, the links of which are in scraping contact with the arcuate bottom. The chains are suspended from arms attached to a horizontal oscillatory shaft.—A. E. D.

*Petroleum oils; Process of desulphurising* — E. B. Cobb, Assr. to Standard Oil Co. U.S.P. (a) 1,357,224 and (b) 1,357,225, 2.11.20. Appl., 13.3.19.

THE oil is freed from elementary sulphur by treatment with (A) an alkaline hydroxide and hydrogen sulphide or (B) an alkaline-earth sulphide.—A. E. D.

(A, c) *Hydrocarbon oils; Apparatus for treatment of* — (a) *Process for treatment of hydrocarbon oils.* R. B. Day, Assr. to A. B. Adams. U.S.P. (a) 1,357,276, (b) 1,357,277, and (c) 1,357,278, 2.11.20. Appl., (A, B) 27.7.18, (c) 30.7.18.

(A) Oil is heated in a coil situated in a distilling chamber which is a continuation of a cracking



chamber heated by gaseous products of combustion. The vapour distilled from the oil passes into the cracking chamber. (n) Oil is fractionated, and the several fractions are cracked by contact with hot inert combustion gases. The heat of the cracked products is utilised to fractionate further quantities of oil. (c) Shale is treated with hot combustion gases whereby oil is first distilled and secondly cracked.—A. E. D.

*Hydrocarbons; Method of and apparatus for cracking* — F. Puening. U.S.P. 1,358,174, 9.11.20. Appl., 21.12.16.

A BODY of oil is maintained in a chamber immediately above a cracking chamber in which there is a number of vertical tubular stills. The oil passes into the stills through pipes extending to the lower end of the latter, and a gas or vapour may be introduced at the lower ends of the pipes to promote circulation or decomposition.

*Hydrocarbons; Furnace for treating* — C. C. Stutz, Assr. to Synthetic Hydrocarbon Co. U.S.P. 1,359,931, 23.11.20. Appl., 30.10.15. Renewed 15.3.19.

A NUMBER of vertical tubes are arranged with their upper ends enclosed within chambers. The chambers are connected at their upper ends with a combustion chamber and at their lower ends with flues leading to a stack. The lower ends of the tubes are similarly enclosed within independent chambers which are connected with an independent combustion chamber and the stack.—J. S. G. T.

*Fuel; Liquid* — J. Black. U.S.P. 1,360,872, 30.11.20. Appl., 18.8.19.

THE fuel consists of 5 galls. of gasoline, 16 oz. of ether, 100 oz. of acetone, 14 oz. of alcohol, and 100 galls. of the distillate obtained by distilling a mixture of an aliphatic hydrocarbon of less than 50° B. (sp. gr. 0.778) with 10% of benzene, the distillate having a higher sp. gr. than the aliphatic hydrocarbon.—L. A. C.

*Coal, tar, heavy oils resulting from the distillation of bituminous coal, etc.; Process of treating* — C. S. Palmer, Assr. to Standard Oil Co. U.S.P. 1,360,973, 30.11.20. Appl., 29.8.17.

THE material is digested, without addition of steam, using a pressure above 4 atm., and a temperature above 200° C., but below that at which sufficient carbonisation occurs to interfere with the process, until the greater portion is transformed into readily volatile products.—L. A. C.

*Coal; Concentration of bituminous — by flotation*. W. J. Mellersh-Jackson. From Metals Recovery Co. E.P. 154,702, 2.9.19.

SEE U.S.P. 1,329,493 of 1920; J., 1920, 220 A.

*Gas from coal and analogous substances; Means to generate* — J. H. Corthesy, Assr. to S. T. S. Castelli. U.S.P. 1,360,503, 30.11.20. Appl., 10.5.19.

SEE E.P. 127,933 of 1918; J., 1919, 565 A.

*Shale and the like; Process of and apparatus for recovering bitumens from* — H. D. Ryan. E.P. 129,992, 16.7.19. Conv., 28.3.18.

SEE U.S.P. 1,327,572 of 1920; J., 1920, 182 A.

*Fuels; Liquid* — U.S. Industrial Alcohol Co., Assees. of A. A. Backhaus. E.P. 140,796, 25.3.20. Conv., 12.10.17.

SEE U.S.P. 1,271,114 of 1918; J., 1918, 539 A.

*Motor-fuel*. A. Hayes, Assr. to U.S. Industrial Alcohol Co. U.S.P. 1,361,153, 7.12.20. Appl., 27.8.19.

SEE E.P. 153,925 of 1919; preceding.

*Coke-chamber and discharging-guide therefor*. L. Wilputte, Assr. to A. A. Wilputte. U.S.P. 1,360,608, 30.11.20. Appl., 4.5.18.

*Gas retorts and the like; Mouthpieces for vertical* — Goodall, Clayton, and Co., Ltd., C. R. Payne, and J. A. Archdale. E.P. 154,758, 6.10.19.

*Gas producers; Fuel feeding and distributing devices for* — H. Nielsen and F. D. Marshall. E.P. 154,307, 21.8.19.

*Regenerative ovens*. U.S.P. 1,361,595. See I.

*Asphalts*. G.P. 325,780. See V.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Acetic acid in pyroigneous acid; Determination of* — V. E. Grotlisch. J. Ind. Eng. Chem., 1920, 12, 1183-1186.

TEN c.c. of the crude acid is distilled with 120 c.c. of xylene and the distillate, after dilution with water, is titrated with N/2 sodium hydroxide solution using phenolphthalein as indicator. The aqueous portion is then separated from the xylene and evaporated to a volume of about 70 c.c., 100 c.c. of water is added, the mixture again evaporated and these operations repeated several times to expel esters, acetone, etc. Other volatile matters are then oxidised by treating the hot solution with permanganate (this does not act on the acetate), the mixture filtered, diluted to 300 c.c., and 200 c.c. of the filtrate is evaporated to 70 c.c., treated with a few drops of concentrated sulphuric acid and hydrogen peroxide to destroy excess of permanganate, a slight excess of barium hydroxide solution is added, the mixture evaporated to a small volume, filtered out of contact with air, and the filtrate containing the acetate then distilled with phosphoric acid in the usual way.—W. P. S.

### PATENTS.

*Coal, shale, or other bituminous substances; Apparatus for the low-temperature distillation of* — K. Prinz zu Löwenstein, A. Irinyi, and T. Kayser. E.P. 144,712, 10.6.20. Conv., 28.10.18.

COAL, shale, or the like is distilled in a double-walled retort heated by steam or hot air passed through the jacket. The retort contains a hollow screw-conveyor through which steam or hot air is also passed from end to end through the blades. The conveyor is automatically scraped by means of an endless band moving over shafts and carrying ribs which project downward between the blades of the conveyor and are moved forward by the rotation of the screw.—W. F. F.

*Volatile hydrocarbons; Apparatus for treating carbonaceous material for the recovery of* — C. C. Bussey. E.P. 154,658, 29.7.19.

IN a continuously acting retort for the low-temperature distillation of coal, lignite, or shale, the heat is supplied entirely by the partial combustion of the material at the bottom of the retort. Clogging of the retort is avoided by the use of a water-cooled ejector bar at the bottom, which removes the coked residue alternately in opposite directions. The temperature is kept low enough to avoid cracking of the distillation products with formation of tar, and the weight of the fuel bed causes the formation of a hard coke even from cannel coal.—C. I.



*Distillation ovens.* G. B. Ellis. From Foundation Oven Corp. E.P. 154,819, 29.1.20.

In a retort for the low-temperature distillation of coal, lignite, or peat, with continuous top feed, the walls of the annular heating flues are made of fire-clay brick in the upper part of the retort and of silica brick in the lower part. Separate exits are provided from appropriate parts of the retort for ammonia-, tar-, and moisture-containing gases, and the temperatures of the different zones are regulated by introducing additional gas or cooling air through a number of holes in the annular heating flues.—C. I.

*Decolorising carbons; Process of making* —. R. W. Mumford, Assr. to Refining Products Corp. U.S.P. 1,359,094, 16.11.20. Appl., 23.3.18.

A stiff pasty mixture containing powdered coal-like matter, tarry matter, and ammoniacal liquor, is charred in a vented retort through which steam is passed to facilitate the removal of vapours, and the temperature is slowly raised to above 600° C.

—J. H. L.

*Mantles for gas lighting by incandescence.* R. H. Stevens. E.P. 154,138, 2.6.20.

To increase the mechanical strength of incandescence mantles they are treated with a thin paste formed of kaolin or like clay mixed with phosphoric acid or with phosphoric acid and aluminium silicate. In the case of inverted mantles, the supporting ring is preferably roughened or provided with holes to facilitate adhesion of the paste, and the paste is applied to the regions around and adjacent to the ring. In the case of upright mantles, the loop and shoulder are coated, preferably on the inner and outer sides. The paste may be applied to mantles in course of manufacture or subsequently.—J. S. G. T.

*Metal filament; Process for heating a* — to any desired temperature. Siemens und Halske A.-G. G.P. 325,644, 18.9.18.

A POSITIVELY charged electrode is arranged in proximity to the filament so that a current passes between it and the filament, and the heating current of the latter is so regulated that a definite relation between the saturation current and the heat energy is obtained. For example, for the production of a standard lamp which will always give approximately the same amount of light, a metal electrode is arranged near to the filament so that a stream of electrons passes between them.—A. R. P.

*Arc lamp carbons; Metal-coated* —. Siemens-Schuckertwerke G.m.b.H. G.P. 325,808, 29.6.19. Conv., 2.10.17.

CATHODE carbons for arc-lamps taking heavy currents are provided with a number of channels or depressions which are completely filled by the metal coating. The latter consists of first, a thin coating of a noble metal, e.g., silver, then a thicker coating of a base metal, e.g., copper.—A. R. P.

### III.—TAR AND TAR PRODUCTS.

*Lignite tar oils; Composition of* —. A. Grün and E. Ulbrich. Z. angew. Chem., 1920, 33, 295–296.

A LIGNITE tar oil (b.p. 180°–260° C.), obtained from a Mond gas producer, after removal of phenols and amines, was distilled with superheated steam; it had b.p. 240°–260° C., sp. gr. at 17.5° C. 0.940, and iodine value (Wijs) 54.84. The bromine addition value was 11.06 and the bromine substitution value 48.78. The oil was then freed from olefines and "naphthylenes" in the usual way, i.e., with a mixture of concentrated and fuming sulphuric acid

and then by means of the formolite reaction; the bromine addition value was thereby reduced to 0, whilst the bromine substitution value was still about 29, showing that saturated hydrocarbons were present which combined with bromine. The iodine value of the oil does not, therefore, represent the amount of unsaturated substances in the oil.

—W. P. S.

*Benzene; Freezing point of wet* — and the influence of drying agents. N. V. Sidgwick. Chem. Soc. Trans., 1920, 117, 1340–1343.

THE freezing point of repeatedly purified, dry benzene is lowered 0.100° C. by saturation with water. On the addition of various dehydrating agents water is withdrawn from the benzene to varying extents depending on the efficiency of the drying agent employed, and the freezing point is correspondingly raised, and a convenient method is thereby afforded for determining the relative efficiency of the drying agents. This efficiency will be the same (at temperatures near 5° C.) for a solution in any solvent in which the drying agent is insoluble. The freezing point of the wet benzene was raised by various drying agents as follows:—sodium sulphate, 0.024° C.; potassium carbonate, 0.061°; copper sulphate, 0.089°; calcium chloride, 0.091°; sodium hydroxide, 0.098°; phosphoric oxide, 0.100° C. The removal of water by phosphoric oxide is therefore apparently complete.—G. F. M.

*Sulphonation; Catalytic action of iodine in* —. I. J. N. Rây and M. L. Dey. Chem. Soc. Trans., 1920, 117, 1405–1407.

IODINE acts as a positive catalyst in sulphonation. Ordinary concentrated sulphuric acid (sp. gr. 1.84) with a trace of iodine can advantageously be used in place of fuming sulphuric acid. The nature of the product is sometimes changed, thus rendering easy the preparation of acids otherwise difficult to obtain. The catalytic sulphonation is facilitated by the presence of an amino-, hydroxy-, or halogen-group, proceeds with less ease with carboxylic acids, and is inhibited by the nitro-group, and probably also by the sulphonio-group, since disulphonic acids are never obtained. There is an optimum temperature for each reaction at which maximum transformation takes place. Among the more noteworthy results toluene gives a 90–95% yield of the *p*-sulphonic acid, benzoic acid a 95% yield of the *ortho*-acid, and chlorobenzene an 85% yield of the *para*-acid.—G. F. M.

*Naphthalene-2.7- and 1.6-disulphonic acids; Method for the detection of* —. J. A. Ambler. J. Ind. Chem., 1920, 12, 1194–1195. (Cf. J., 1920, 815 A.)

THE dry acetone-insoluble  $\beta$ -naphthylamine salts of the acids are boiled with a small quantity of a mixture of 95% alcohol, 4, and water, 1 vol., the mixture filtered, and the filtrate cooled; the  $\beta$ -naphthylamine salt of the 2.7-disulphonic acid crystallises out. Another portion of the dry salts is boiled with water and titrated with *N*/1 sodium hydroxide solution, using phenolphthalein as indicator; the precipitated amine is filtered off, and the filtrate evaporated to dryness. The dry residue is dissolved in twice as much water as the volume of sodium hydroxide solution used, and the solution is treated with its own volume of concentrated sulphuric acid; on cooling, the sodium salt of the 1.6-disulphonic acid crystallises out gradually (after some hrs.) whilst the 2.7-acid remains in solution.

—W. P. S.

*Methyl derivatives of the xyldines and naphthylamines; Preparation of* — by catalysis. A. Mailhe and F. de Godon. Comptes rend., 1920, 171, 1154–1155.

WHEN a xyldine or naphthylamine is mixed with

methyl alcohol and passed in the form of vapour over alumina at 360°–380° C., in all cases a mixture of methyl and dimethyl derivatives is obtained, the whole of the primary base being methylated.—W. G.

*Distillation of tar.* Offermann. See IIa.

*Toluene from turpentine.* Mahood. See XX.

*Phenols.* Fuchs and Elsner. See XXI.

#### PATENTS.

*Hydrogenation apparatus.* The Dayton Metal Products Co., Assees. of T. Midgley, jun. E.P. 150,991, 7.5.19. Conv., 5.10.18.

CLAIM is made to apparatus suitable for hydrogenating benzene as described in E.P. 133,666 (J., 1920, 717 A).—L. A. C.

*Treating tar oils.* U.S.P. 1,360,973. See IIa.

### IV.—COLOURING MATTERS AND DYES.

*ar-Dihydro- $\alpha$ -naphthylamines and their derivatives. Studies in the dihydronaphthalene series.* I. F. M. Rowe and E. Levin. Chem. Soc. Trans., 1920, 117, 1574–1579.

THE most suitable compounds to prepare for the purification and characterisation of  $\alpha$ -naphthylamine, its two dihydro-derivatives, and its tetrahydro-derivative are the benzylidene derivatives, which are readily obtained in a pure condition from each of the crude bases, and which, on hydrolysis, yield the pure bases. The dihydro- $\alpha$ -naphthylamines condense with diazonium salts, with the direct formation of aminoazo-compounds, and they may also be diazotised and combined with amines or phenols, forming azo-dyes, which differ in shade from similar azo-dyes derived from  $\alpha$ -naphthylamine or *ar*-tetrahydro- $\alpha$ -naphthylamine. The shade produced by an azo-derivative of 5,8-dihydro- $\alpha$ -naphthylamine is more nearly related to that produced by a similar azo-derivative of  $\alpha$ -naphthylamine, whilst the shade produced by a 7,8-dihydro- $\alpha$ -naphthylamine derivative is more nearly related to that produced by a similar tetrahydro- $\alpha$ -naphthylamine derivative.—F. M. R.

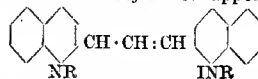
*Carbazole Blue and Carbazole Violet.* M. Copisurrow. Chem. Soc. Trans., 1920, 117, 1512–1550.

CARBAZOLE Blue, obtained by fusing carbazole with oxalic acid, is a tricarbazylmethane colouring matter of formula,  $(C_{12}H_7NH)_3C:C_{12}H_7NH.CO_2H$ , and it may reasonably be assumed that the methane carbon atom is linked to each carbazole nucleus at the *p*-position with respect to the nitrogen atom. Carbazole Blue, like the triphenylmethane dyes, furnishes a carbinol which gives with acids variously coloured salts, of which the formate is identical with the colouring matter. The corresponding tricarbazylmethane, prepared by reducing Carbazole Blue, forms white, rhombic needles, m.p. 217°–218° C. When 9-ethylcarbazole is fused with oxalic acid, a violet colouring matter (Carbazole Violet) is obtained, and the carbinol and parent hydrocarbon of this compound have also been prepared. Both Carbazole Blue and Carbazole Violet are readily sulphonated, and consequently the presence of a phenylene group, like that of the benzyl and phenyl groups, makes possible the production of soluble dyes of the triphenylmethane series.—F. M. R.

*Cyanine dyes. III. Constitution of Pinaeyanol.* W. H. Mills and F. M. Hamer. Chem. Soc. Trans., 1920, 117, 1550–1562.

PINAEYANOL (E.P. 16,227 of 1905; J., 1906, 368) is an example of the class of blue sensitising dyes for

which the name carbocyanines has been proposed. The structural formula advanced by O. Fischer (J., 1919, 199a) does not appear to represent the composition of these dyes correctly, as it contains one atom of carbon less than is indicated by the analytical results, whilst the representation of the carbocyanines by Wise, Adam, Stewart, and Lund (J., 1919, 456a) as dimethyl derivatives of the true cyanines is not in accord with their photosensitising action, nor does it agree with their behaviour on oxidation. The most probable structure of the carbocyanines appears to be:



In the case of Pinaeyanol, R represents the ethyl radicle. There thus appear to be three types of cyanines, the true cyanines, the isocyanines, and a third type, at present unknown in the quinoline series, of which the carbocyanines are an example in which the :CH. group connecting the two quinoline nuclei has been replaced by the chain :CH.CH:CH.—F. M. R.

*Quinocyanines. II.* O. Fischer and G. Scheibe. J. prakt. Chem., 1919, 100, 86–90.

By the action of 4-phenylquinolymethiodide on the  $\Psi$ -base of 2-methyl-4-quinolymethiodide a red dye-stuff is produced, which is identical with that previously obtained by treatment of the  $\Psi$ -base in ethereal solution with air and carbon dioxide (J., 1919, 199a), and is therefore 4,4'-diphenyl- $\Psi$ -isocyanine. 4,4'-Diphenyl-6,6'-dimethyl- $\Psi$ -isocyanine is obtained by the second method from 2,6-dimethyl-4-phenylquinolymethiodide. The parent  $\Psi$ -isocyanine is prepared by the addition of potassium hydroxide in excess to a solution of 2-iodoquinolymethiodide and quinaldylmethiodide in hydrochloric acid. (Cf. J.C.S., Jan.)—J. K.

*"Furo Green."* O. Fischer and L. Grahl. J. prakt. Chem., 1920, 100, 159–167.

THE leuco-base of Furo Green may be obtained in 30% yield by the condensation of furfural with dimethylamine in presence of zinc chloride, and is converted into the dyestuff by means of lead peroxide. "Furo Green Ae" is similarly obtained by the use of diethylaniline. These dyestuffs dye silk, cellulose, and wool a brilliant yellowish-green, but in the last case the fastness to light is only slight (Cf. J.C.S., Jan.)—J. K.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Methyl alcohol from lignin.* E. Heuser and H. Schmelz. Cellulose Chemie, 1920, 1, No. 6. Paper, Nov. 17, 1920, 25, 32, 50.

ALL the methoxyl groups of lignin are not removed by the bisulphite treatment of wood for the isolation of cellulose, nor by the hydrolysis of wood by dilute acids for the saccharification of the cellulose, whilst the lignin obtained as a by-product of Willstätter and Zechmeister's method of hydrolysis in the cold with concentrated hydrochloric acid (J., 1913, 822) still retains almost the entire methoxyl content of the original wood. This lignin by-product, containing on the average 12–13% of methoxyl, is therefore a valuable potential source of methyl alcohol. By dry distillation the greater part of the methoxyl groups is destroyed, but much more satisfactory results are obtained by hydrolytic methods. Distillation with dilute hydrochloric acid (57%) gave acetic acid 2.53%, acetone 0.13%, and methyl alcohol 2.50%. Steam distillation in presence of hydrochloric acid was less satisfactory,

giving acetic acid 0.47%, acetone 0.53%, and methyl alcohol 1.04%. By far the best results, approximately quantitative in respect to methyl alcohol, were obtained by heating for 2 hrs. with dilute hydrochloric acid under 5–6 atm. pressure, this process being repeated with the residue from the first treatment. The total yield of products was acetic acid 2.19%, acetone 0.73%, and methyl alcohol 14.83%. The demethylated residue gave only a faint turbidity with silver nitrate by Zeisel's test.—G. F. M.

#### PATENTS.

*Flax; Retting* —. B. S. Snimmers. U.S.P. 1,360,329, 30.11.20. Appl., 2.12.18.

FLAX is steeped in a bath of retting liquor and the insoluble products only are removed from the bath.—A. J. H.

*Bast fibres of mallows; Process for separating the* —. H. Küchenmeister. G.P. 325,886, 20.12.18.

MALLOW stalks are heated with alkaline liquors, the layers of bast fibres are stripped off, and the fibres separated and freed from mucilaginous and insoluble substances by a thorough washing. For *Althaea cannabina* 8%, and for *Sida alba* 4% caustic soda is used. Fibres to be used for fine fabrics should be boiled twice with alkali.—A. J. H.

*Shrinking fabrics; Method for* —. W. Dahse. G.P. 325,797, 25.2.19.

THE fabrics are treated with strong caustic soda or other substances with a similar action, to which may be added substances which diminish the shrinkage, e.g., glycol or glycerin.—A. J. H.

*Cellulose; Manufacture of — from straw, wood, and other cellulose substances.* H. Oexmann. G.P. (A) 306,325, 31.7.17, and (B) 309,259, 25.5.18.

(A) BEFORE treatment with strong alkalis, the raw materials are treated with solutions of weak alkalis, e.g., lime. 100 kg. of straw is boiled first with 400–600 l. of water and 5–6 kg. of quicklime, and then with 4 kg. of caustic soda. The product is equal to that obtained by using 9–10 kg. of caustic soda only. (B) The treatment with the weak or with the strong alkalis or both may be effected at a temperature below boiling point.—A. J. H.

*Half-stuff for paper making, cellulose, artificial silk, etc.; Manufacture of* —. K. Scholz, B. P. von Ehrenthal, and M. von Halle. G.P. 325,918, 21.3.17.

FIBROUS material as hemp, bagasse, tobacco stalks, jute, bamboo, etc., is treated by the usual method employed in digesting flax by the sulphite process, except that the alkali content of the sulphite liquor is increased to three or four times the usual amount, and is then digested with alkali.—A. J. H.

*Cellulose acetate; Manufacture of compositions, preparations, or articles having a basis of* —. H. Dreyfus. E.P. 154,334, 26.8.19.

ADDITIONAL aromatic sulphonamide compounds (cf. E.P. 132,283 and 133,353; J., 1919, 896 A; 1920, 14 A), having high boiling points and insoluble in water, which are suitable as solvents or the like in the manufacture of dopes, films, cellinoid-like masses, artificial silk, etc., having a basis of cellulose acetate are: benzenemonomethylsulphonamide (b.p. about 180° C. at 1 mm.), benzenemethylethylsulphonamide (b.p. 170°–175° C. at 1–2 mm.), mixtures of *o*- and *p*-toluenedimethylsulphonamides (b.p. 133°–137° C. at 1 mm.), mixtures of *o*- and *p*-toluenemethylethylsulphonamides (b.p. 188°–189° C. at 1 mm.), mixtures of *o*- and *p*-toluenediethylsulphonamides (b.p. 136°–142° C. at 1 mm.), *o*-toluenedimethylsulphonamide (b.p. 148°–152° C. at 2 mm.), *o*-toluenediethyl-

sulphonamide (b.p. 152°–155° C. at 2 mm.), mixtures of isomeric xylenedimethylsulphonamides (b.p. 150°–155° C. at 1 mm.), mixtures of isomeric xylenediethylsulphonamides (b.p. 148°–154° C. at 1 mm.), mixtures of isomeric xylenemethylethylsulphonamides (b.p. 155°–156° C. at 1 mm.). These products are all liquid at the ordinary temperature and form jellies on cooling. For some applications, these aromatic dialkylated sulphonamide derivatives are not so good as benzenemonomethylsulphonamide and the monoalkylated sulphonamides specified in E.P. 132,283 and 133,353 (*loc. cit.*). The new solvents may be employed with any usual solvents or substances such as triphenyl phosphate, tricresyl phosphate, and acid-nouralising aliphatic derivatives of urea which are liquid or have a low melting point.—A. J. H.

*Cellulose-ester composition.* J. M. Kessler, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,360,759, 30.11.20. Appl., 19.6.20.

THE composition consists of nitrocellulose and an acetin free from mono-acetin and free acetic acid.—A. J. H.

*Paper or other materials in sheet form; Apparatus for drying* —. O. Minton. E.P. 154,120, 26.3.20.

PAPER is dried by passing it without tension through a liquid seal into a vacuum chamber, over numerous rollers, some of which are immersed in a heated neutral fluid, and out through the same or a different liquid seal. Mercury may be used for the sealing liquid and for the heated neutral fluid. Paper having "cockled" effects may also be produced by this method, and the drying temperature may be low or high.—A. J. H.

*[Sized] paper manufacture.* R. Jennison and J. P. de Chanaz. E.P. 154,366, 9.9.19.

WOOD pulp or similar cellulosic material is treated with 20% of starch and 1 to 5% of caustic soda and beaten for  $\frac{1}{2}$  to 8 hrs. The caustic soda is neutralised by the addition of alum or other aluminium salt, the pulp is diluted with water, passed through a paper-making machine, and the paper dried. For transparent grease-proof paper, the paper web is incompletely dried, lightly calendered, and then friction-calendered until transparent. If more than 20% of starch be used, the strength and transparency of the paper are increased.—A. J. H.

*Paper pulp; Process of making* —. R. H. McKee and A. A. Holmes. U.S.P. 1,360,658, 30.11.20. Appl., 24.5.19.

WOOD which contains resin is subjected to the action of liquid sulphur dioxide and is then converted into pulp by the usual methods.—A. J. H.

*Asphalts [for sizing paper, fabrics, etc.]; Means for lowering the melting point of* —. Chem.-analyt. Lab. Schick & Horák. G.P. 325,780, 7.7.18.

THE melting point of asphalt is lowered by the addition of a terpene or an aliphatic or aromatic alcohol. For the impregnation of yarn, fabric, paper, cardboard, etc., asphalt containing 0.1–1% of fusel oil is suitable.—A. J. H.

*Paper; Process of re-working old* —. J. M. Burby. E.P. 154,670, 22.8.19.

SEE U.S.P. 1,327,590 of 1920; J., 1920, 264 A.

*Sulphite [cellulose] liquor; Process for the production of valuable organic and inorganic substances from waste* —. R. W. Strehlenert. U.S.P. 1,361,506, 7.12.20. Appl., 10.5.17.

SEE G.P. 308,144 of 1917; J., 1918, 764 A.

*drying apparatus for varnished or painted paper.*  
J. W. Wilson. E.P. 154,764, 15.10.19.

*concentrating sulphite-cellulose waste lyes.* E.P.  
154,355. See I.

*drying cylinders.* E.P. 154,508. See I.

*Fermentable sugars.* U.S.P. 1,358,998. See XVIII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Cotton dyed with sulphur colours; After-treatment of — [with copper sulphate].* A. S. Eieblin.  
Text. Col., 1920, 42, 777—778.

BLEACHED cotton was boiled with 30% solution of sodium sulphide containing sodium carbonate, thoroughly washed, and treated with 3% of copper sulphate and 1½% of acetic acid. It absorbed 0.04% of copper. Under similar circumstances, unbleached cotton absorbed 0.1% Cu. The following amounts of copper were fixed by cotton dyed with various sulphur dyes:—5% Sulphur Olive O. (Metz), 0.20% Cu; 10% Sulphur Olive O. (Metz), 0.36%; 5% Thiogene Blue B. (M.L.B.), 0.04%; 5% Thiogene Orange R. (M.L.B.), 0.04%; 5% National Sulphur Blue B.D., 0.06%; 5% National Sulphur Blue L., 0.06%; 5% Thiogene Yellow 5 G. conc., 0.17%; 5% Thiogene Gold Yellow A., 0.18%; 5% Thiogene Yellow G.G., 0.16%; 5% Thiogene Yellow G.H., 0.15%; 5% Thiogene Deep Blue 2 R.L. conc., 0.03%; 5% Thiogene Brown 3 R., 0.07%; 5% Katigen Yellow Brown (Bayer), 0.08% Cu.

—A. J. H.

### PATENTS.

*Fabrics; Method of dyeing and preserving —.*  
C. L. Cole (C. H. Cole extrix.). U.S.P. 1,361,130,  
7.12.20. Appl., 6.6.17.

TEXTILE fabrics are treated with a solution of tannic acid and are subsequently immersed in a bath formed by suspending calcium carbonate in a solution of copper sulphate.—A. J. H.

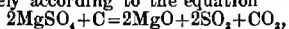
*Fireproofing and/or waterproofing treatment of materials.* A. Arent. E.P. 132,813, 20.9.19.  
Conv., 20.9.18.

FABRICS are rendered fireproof by treatment with a solution of an antimony or bismuth salt (preferably antimony trichloride) in a volatile carbon chloride solvent such as carbon tetrachloride or chloroform, evaporating off the solvent, and hydrolysing the remaining antimony salt by exposing the fabric to steam or water. If the fireproofing solution contains a vulcanisable gum of the caoutchouc type or gutta-percha, the fabrics are also rendered waterproof. A solution of anhydrous antimony tri-iodide in carbon bisulphide may also be used.—A. J. H.

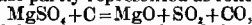
## VII.—ACIDS; ALKALIS; SALTS; NON- METALLIC ELEMENTS.

*Sulphur and sulphuric acid; Preparation of — from alkaline-earth sulphates.* E. H. Riesenfeld.  
J. prakt. Chem., 1920, 100, 115—158.

THE reduction of kieserite by carbon does not take place solely according to the equation



but is at least partly represented as follows:—



because sulphur is always found in the residue when less than one atomic proportion of carbon is employed. The best results are obtained with the

proportion 1:1 at 750°–850° or 1.5:1 at 950° C., when the sulphur is almost entirely converted into sulphur dioxide, with a little free sulphur. Greater proportions of carbon do not produce the favourable effect which might be expected. The reduction of gypsum and anhydrite by carbon takes place fairly rapidly from 700° C. upwards approximately according to the equation  $\text{CaSO}_4 + 3\text{C} = \text{CaS} + \text{CO}_2 + 2\text{CO}$ . Strontium and barium sulphates behave similarly. The equilibrium  $\text{CaS} + \text{H}_2\text{O} + \text{CO} \rightleftharpoons \text{CaCO}_3 + \text{H}_2\text{S}$  cannot be utilised for the disposal of the calcium sulphide obtained as above, since temperatures, e.g., 700° C., at which it is sufficiently rapidly attained, very largely favour the formation of calcium sulphide. At 800°–1000° C. the reaction,  $\text{CaSO}_4 + \text{CH}_4 = \text{CaS} + \text{CO}_2 + 2\text{H}_2\text{O}$ , would probably permit complete reduction, if sufficiently prolonged, but below 800° C. dehydration alone occurs. Above 1100° C. some oxide is produced, probably according to the equation,  $\text{CaS} + \text{H}_2\text{O} = \text{CaO} + \text{H}_2\text{S}$ , and, as would therefore be expected, excess of steam at 1200°–1300° C. favours complete removal of the sulphur. This is, however, obtained almost entirely as sulphur dioxide and sulphur—chiefly the latter when only a slight excess of steam is used—partly owing to dissociation of hydrogen sulphide and partly owing to its reaction with steam. The amounts of sulphur dioxide and sulphur produced by the action of steam on calcium sulphide agree with the above statement. Further, calcium sulphate is converted directly into the oxide by treatment with water and steam at 1200°—or, more than six times more rapidly, at 1300°. Owing to the reduction of sulphur dioxide by carbon monoxide and hydrogen, sulphur is the chief product, only 50% of sulphur dioxide being obtained even when 850 times the theoretical proportion of steam is employed.—J. K.

*Nitrogen in nitrates and nitrites; Determination of — by copper-magnesium.* T. Arnd. Z.  
angew. Chem., 1920, 33, 296—298.

IN a method described previously by the author (Z. angew. Chem., 1917, 30, 169), in which the nitrite or nitrate is reduced to ammonia by distilling with copper-magnesium alloy and magnesium chloride solution, an excess of the latter is required to prevent the interference of sulphates and other salts. At least 2.5 g. of the alloy must be used for each 0.5 g. of nitric nitrogen present.—W. P. S.

*Potash in wood ashes and treater dust; Modified method for the determination of water-soluble —.* H. D. Haskins. J. Assoc. Off. Agric.  
Chem., 1920, 4, 82—84.

FRESHLY made dry ashes and dry treater dust will yield appreciably more water-soluble potash if allowed to remain in contact with water for several hours previous to extraction with boiling water. In the case of ash products which contain a considerable amount (12–30%) of water very little advantage results from the preliminary soaking in water.—W. G.

*Potassium platinichloride; Composition of —.*  
A. Vürtheim. Chem. Weekblad, 1920, 17, 637—640.

POTASSIUM platinichloride reaches constant weight after drying for 2 hrs. at 150° C., but this treatment is not enough to remove all the enclosed water, and precipitation from aqueous solution by means of alcohol and drying at 110° C., gives results which agree better with the formula  $\text{K}_2\text{PtCl}_6$ . The accuracy of the accepted atomic weight of platinum is questioned. (Cf. J.C.S., Jan.)—S. I. L.

*Leucites and leucitic minerals; Analysis of —.*  
G. Tommasi. Ann. R. Staz. Chim.-Agrar.  
Sperim., 1917-1919, II., 9, 95—106.

1.25 GRAM of the leucite or leucitic mineral, ground

to an impalpable powder, is treated in a covered beaker with about 45 c.c. of hydrochloric acid and 5 c.c. of nitric acid, first for 2 hrs. on a water-bath and then for 20 mins. on a sand-bath. The insoluble silicious matter is then estimated in the ordinary way, the alkalis are isolated as usual, and the potassium estimated by the perchlorate method. This procedure gives results agreeing with those obtained when the mineral is attacked by hydrofluoric acid or by ammonium chloride and calcium carbonate according to the Lawrence Smith method.—T. H. P.

*Electrolytes; Flocculating power of —.* (Opacity number.) N. Bach. J. Chim. Phys., 1920, 18, 46–64.

FROM experiments on colloidal ferric hydroxide and arsenic trisulphide, the author concludes that the power of electrolytes to effect the flocculation of colloidal sols may be determined accurately by dropping the electrolyte solution at intervals of precisely 20 secs. into a measured volume of the sol contained in a glass cylinder of 20 c.c. capacity and observing the amount of electrolyte required to produce a definite turbidity. The opacity number is the inverse of the amount of electrolyte required; that of potassium chloride is taken as unity. The relative opacity numbers of two electrolytes depend solely on the nature of the colloidal sol, and may even be independent of the chemical nature of the sol. The flocculating powers of the alkali-ions and halogen-ions decrease in the following order: Cs, Rb, NH<sub>4</sub>, K, Na, Li and Cl, Br, I. Other things being equal, any ion will flocculate an oppositely charged sol or protect a similarly charged one in proportion to the amount of charge on the ion. The flocculating power is much greater than the protective power. In the case of halogen ions, the differences between the potentials required to discharge them are proportional to their flocculating power. When various electrolytes have a common ion their flocculating powers are additive. Colloidal sols are extremely sensitive to traces of polyvalent impurities. Divalent ions have about 30 to 40 times the flocculating power of univalent ions, and trivalent ions have about 12 times the flocculating power of divalent ions.—A. B. S.

*Cupric hydroxide; Solubility of — in concentrated sodium hydroxide solution.* E. Müller. Z. angew. Chem., 1920, 33, 303–305.

CONCENTRATED sodium hydroxide solution dissolves cupric hydroxide, yielding a violet-blue, unstable solution; in the case of 48% sodium hydroxide solution the copper content may amount to about 3%. The author discusses the matter from a physico-chemical point of view, and concludes that the solution of the copper hydroxide is due to the formation of complex ions.—W. P. S.

*Carbonyl chloride; Preparation and physical properties of —.* R. H. Atkinson, C. T. Heycock, and W. J. Pope. Chem. Soc. Trans., 1920, 117, 1410–1426.

CARBONYL chloride is most conveniently prepared in quantity by the method, originally described by Paternò (Gaz. Chim. Ital., 1878, 8, 233), of passing chlorine and carbon monoxide over charcoal. The wood charcoal used in the Army box respirator proved the most efficient for the purpose, effecting complete and rapid conversion of the mixed gases into carbonyl chloride at ordinary temperatures, 50° C. below that at which it began to show catalytic activity towards a mixture of hydrogen and chlorine—a fact of importance when carbon monoxide containing hydrogen is used. Dissociation of carbonyl chloride begins at temperatures above 200° C.; at 309° C. it amounts to 5.61%, and at

503° C. 55–56%. The vapour pressure of carbonyl chloride was determined between 100° C. and –189° C. The vapour pressure curve begins to fall rapidly only below –40° C., hence considerable losses will occur in the manufacture unless the effluent gas is either cooled below this temperature or scrubbed with a suitable solvent, such as toluene, xylene, or chlorobenzene, in which the gas is readily soluble. Carbonyl chloride freezes at –126° C., its sp. gr. at 0° C. is 1.435, and the mean coefficient of cubical expansion between –79° and +49.9° C. is 0.00177.—G. F. M.

*Carbon dioxide; Preparation of pure —.* R. C. Farmer. Chem. Soc. Trans., 1920, 117, 1446–1447.

THE difficulty of preparing carbon dioxide free from air, even when all the solutions used in making the gas are previously boiled, is overcome by using solutions of potassium bicarbonate and sulphuric acid, each of which is freed from air by bubbling carbon dioxide through it before the two are brought together. A convenient generator for the purpose consists of a Woulff's bottle, into two necks of which pear-shaped funnels with long wide stems, containing the respective solutions, are fitted, a narrow extension passing down to dip just beneath the surface of a layer of mercury in the bottom of the bottle for the purpose of preventing back diffusion. A branched gas-delivery tube is fitted into the third neck of the Woulff's bottle from one arm of which the gas is drawn off as required through a glass stop-cock, whilst the other arm leads to the top of the pear-shaped funnels, and a tube passed down the stems of each so as to bubble the gas through the columns of liquid. The flow of the reagent solutions into the bottle is automatically regulated by the gas delivery stop-cock; when this is shut, the back pressure prevents any further quantity of solution entering the bottle. Once started the apparatus need never be disconnected, and spent liquor can be drawn off by means of a glass tube passing through the neck to just above the surface of the mercury.—G. F. M.

#### PATENTS.

*Acids [e.g., nitric acid]; Production of —.* C. L. Tayntor. U.S.P. 1,361,416, 7.12.20. Appl., 29.11.19.

SULPHURIC acid is added to sodium nitrate in a number of generating stills, delivering into a common expansion chamber, which in turn is connected with several "bleachers" and condensers arranged in parallel, and communicating with a common scrubbing tower.—C. L.

*Potassium sulphate and hydrochloric acid; Manufacture of —.* Fabr. de Prod. Chim. de Thann et de Mulhouse. E.P. 154,111, 27.2.20. Conv., 4.12.19. Addn. to 137,296 (J., 1920, 516 A).

IN the process described in E.P. 137,519 (J., 1920, 689 A), the three stages of desiccation, heating at 300° C., and heating at 700°–800° C., may take place in a single direct-flame furnace, or in a compound direct-flame and muffle-furnace; instead of operating in three stages the process may be conducted progressively in a single furnace, care being taken to keep the mass solid and porous. Aqueous sulphuric acid containing more than 70–72% SO<sub>3</sub> may be used, the proportions being such as to retain the pulverulent nature of the mixture.—W. J. W.

*Sulphate of ammonia; Manufacture of — and apparatus therefor.* N. Wilton. E.P. 154,328, 26.8.19.

WET ammonium sulphate from the saturator is thrown into a tilting vessel fitted with a lid and

mounted on trunnions, and having a perforated false bottom. The adherent mother-liquor and most of the free acid are then removed by blowing in dry steam at 70–80 lb. pressure, with or without air. The remaining free acid may be neutralised by introducing ammonia gas. The vessel is tilted to the side opposite to the saturator to remove the dried salt.—C. I.

*Soda; Process of manufacturing* —. T. Nishigawa. U.S.P. 1,359,097, 16.11.20. Appl., 30.9.19.

In the ammonia-soda process the mother liquor from which sodium bicarbonate has been separated is treated with sodium chloride, and then cooled and treated with sufficient ammonia gas to convert the bicarbonates still in solution into normal carbonates, whereupon ammonium chloride separates out. The residual mother liquor is again used as brine for the production of sodium bicarbonate.

—J. H. L.

*Manganese-containing bodies [manganese chloride]; Producing* —. A. A. Wells, Assr. to National Carbon Co. U.S.P. 1,359,640, 23.11.20. Appl., 29.6.18.

THE spent mixture from dry batteries is treated with hydrochloric acid, carbonaceous material is filtered off, and the filtrate boiled to drive off chlorine and excess acid. The solution is digested with freshly precipitated manganese carbonate to precipitate iron compounds and filtered.

—J. S. G. T.

*Magnesium chloride and the like; Method of making* —. E. O. Barstow, Assr. to The Dow Chemical Co. U.S.P. 1,359,782, 23.11.20. Appl., 1.3.18.

SLAKED dolomitic lime is treated with sulphur dioxide and carbon dioxide, and calcium chloride is added to the product.—W. J. W.

*Phosphate; Process for the manufacture of* —. G. R. Brobst. U.S.P. 1,360,248, 23.11.20. Appl., 26.3.19.

A MIXTURE of phosphate rock, felspar, limestone, iron ore, furnace slag, and sodium carbonate is calcined, and the volatile products are collected with the aid of a spray of dilute ammonia.—W. J. W.

*Carbonyl chloride; Process of making* —. S. Peacock. U.S.P. 1,360,312, 30.11.20. Appl., 29.10.19.

CARBONYL chloride is produced by passing a mixture of air and chlorine over heated carbonaceous material.—D. F. T.

*Molybdenum compounds; Production of* —. G. Richter, Assr. to The Chemical Foundation, Inc. U.S.P. 1,360,581, 30.11.20. Appl., 12.7.17. Renewed, 19.4.20.

A MATERIAL containing molybdenum is treated with a sulphurising agent and water, and the resulting solution treated with a desulphurising agent.

—A. B. S.

*Permanganate; Process for electrolytic production of* —. R. E. Wilson and W. G. Horsch. U.S.P. 1,360,700, 30.11.20. Appl., 29.11.18.

AN anode containing manganese is employed in the continuous electrolysis of an electrolyte consisting of a solution of an alkali carbonate as anolyte and a caustic alkali solution as catholyte. The solution of alkali carbonate is added continually and the electrolyte is kept in circulation.—J. S. G. T.

*Oxides in neutral water solutions; Electrolytic production of* —. S. Fry. U.S.P. 1,361,041, 7.12.20. Appl., 13.11.16.

HYDRATED oxides are produced direct from metals by employing the metal as anode in the electrolysis of an aqueous solution of a salt of sodium, potassium, ammonium, or calcium, of such concentration that the metal is deposited as hydrated oxide.

—J. S. G. T.

*Acid ammonium phosphate; Process for the preparation of* —. Gewerkschaft ver. Constantin der Grosse. G.P. 307,093, 20.5.14.

AMMONIA is passed into a solution of phosphoric acid of 30° B. (sp. gr. 1.26) until the specific gravity rises to 1.28 (32° B.), whereby acid ammonium phosphate is precipitated.—A. R. P.

*Combustion of nitrogen; Process for* —. Process for the separate preheating of reacting gases, especially for combustion of nitrogen. Gewerkschaft des Steinkohlenbergwerks "Lothringen," and M. Ketting. G.P. (A) 325,800, 28.10.19 (Addn. to 324,264; J., 1920, 783A), and (B) 326,228, 31.12.19.

(A) THE finely divided carbon added to the reacting gases in the combustion of nitrogen in a hydrogen or carbon monoxide flame is introduced into the gases by passing them through a preheater over heated carbon compounds, e.g., fused anthracene. (B) The reacting gases (heating gas and air) are passed in layers through the narrow channels of a preheater into the combustion chamber. The hot issuing gases from the latter serve to preheat further quantities of the gases.—A. R. P.

*Kieserite; Separation of* — from a mixture of potassium chloride and kieserite suspended in a liquid. Kaliwerke Grossherzog von Sachsen A.-G., and K. Hepke. G.P. 326,156, 9.4.18.

THE liquid in which a mixture of kieserite and potassium chloride is suspended is passed through an open channel or closed tube into a long, deep, and narrow trough provided with a conveyor belt running in the opposite direction to the flow of liquid. The kieserite alone settles. The finer and slimier the kieserite is, the longer and deeper the trough should be.—A. R. P.

*Salts of trivalent chromium; Process for the preparation of* —. Chem. Fabr. vorm. Weiler-ter Meer. G.P. 326,268, 17.11.18.

WASTE gases containing sulphur dioxide, e.g., the issuing gases from the manufacture of sulphuric acid, are passed through a solution of chromic acid or an acidified solution of its salts.—A. R. P.

*Chlorides; Process and apparatus for electrolytic decomposition of* —. B. Cataldi. E.P. 127,255, 20.5.19. Conv., 20.5.18.

SEE U.S.P. 1,336,281 of 1920; J., 1920, 405 A.

*Nitrogen; Method and apparatus for the fixation of atmospheric* —. J. S. Island. E.P. 154,356, 3.9.19.

SEE U.S.P. 1,316,445 and 1,317,705 of 1919; J., 1919, 819 A, 901 A.

*Sulphate of ammonia; Manufacture of neutral* —. P. Parrish and W. A. M. Valon, Assrs. to South Metropolitan Gas Co. U.S.P. 1,360,785, 30.11.20. Appl., 11.8.19.

SEE E.P. 141,819 of 1919; J., 1920, 447 A.

*Hydrogen; Manufacture of* —. S. W. Bray and I. H. Balfour, Assrs. to British Oxygen Co. U.S.P. 1,360,876, 30.11.20. Appl., 31.3.20.

SEE E.P. 144,751 of 1919; J., 1920, 571 A.

## VIII.—GLASS; CERAMICS.

*Glass; Annealing temperature of* — F. Weidert and G. Berndt. *Z. tech. Phys.*, 1920, 1, 51—58. *Chem. Zentr.*, 1920, 91, IV., 642—643.

THE annealing temperature of a glass may be determined by measuring the difference between the refractive indices of the ordinary and extraordinary rays of light passed through the glass before and after cooling from various temperatures between 200° and 600° C., and plotting this difference against the temperature of the glass before cooling. The graph so produced is a hyperbolic curve with a sharp bend at the annealing temperature. This point is independent of the size of the specimen and of the rate of cooling within wide limits. The deformation temperature (i.e., the temperature at which a 25-mm. cube set on one of its corners on a specimen of the polished glass and half embedded in kieselguhr makes an appreciable impression on the glass after 6 hrs. heating) is about 30° C. below the temperature at which two plates of the glass permanently cohere, and 40°—50° C. higher than the annealing temperature of flint glasses and 70°—100° C. higher than the annealing temperature of crown and barium glasses made at the Sendling works. The deformation temperature of a glass is characteristic, but is also a function of the duration of the heating and of the size of the specimen.

—A. B. S.

*Lead glasses; Composition of* — R. J. Montgomery. *J. Amer. Ceram. Soc.*, 1920, 3, 900—912.

THIRTY-TWO lead glasses are discussed, the compositions being expressed as  $x\text{RO}, 1\text{SiO}_2$ ,  $x$  lying between 0.30 and 1.22, and the refractive index varying from 1.51 to 1.95. The influence of RO content and PbO in RO content on refractive index and dispersive power are shown graphically. A linear relationship connects the refractive index and the PbO in the RO content. Some lead glasses contain free silica, others consist of lead silicates containing no alkali but having an excess of lead oxide, which the lead silicate must take into solution to give a glass.—H. S. H.

*Clays of low plasticity and strength; Effect of wet grinding, screening, electrolytes, and dextrin on* — H. W. Douda. *J. Amer. Ceram. Soc.*, 1920, 3, 885—892.

THE dry strength of clays is increased by wet grinding for 2 hrs., additional increases being obtained by adding 1% of caustic soda and wet grinding, screening through a 150-mesh sieve, or adding 1% of dextrin. The plasticity of the clays is increased by wet grinding, screening, and the addition of 1% of dextrin. Caustic soda causes the clay to become tougher and more difficult to mould. The drying shrinkage is increased, the apparent density decreased, and the water of plasticity increased by wet grinding. The fineness of grain of the clay is increased by wet grinding and further increased by adding 1% of caustic soda. The strength of the clays after burning to cone 2 is increased by the above treatments.—H. S. H.

*Heat transmission of brick and high-temperature insulating materials.* R. A. Horning. *J. Amer. Ceram. Soc.*, 1920, 3, 865—876.

A TEST-PIECE was heated electrically by coils of wire in contact with it. The cool end rested on a water-cooled plate, baffles causing the water to flow in a zigzag path across the plate. Only the heat passing through the central part of the heated area (about 1/9 of the whole area) was measured, the quantity being obtained from the current and fall of voltage in the central heating coil. The temperature of the hot face was given by a thermo-couple embedded in

it, and the average temperature of the water entering and leaving the cooling plate gave the temperature of the cool face. By replacing the cooling plate by electrically heated coils of wire, a constant difference of temperature was maintained between the faces and the heat transmission measured for different temperatures of the hot face. The materials examined, arranged in order of decreasing thermal conductivity, were magnesite brick, silica brick, vitrified building brick, firebrick, re-pressed burned kieselguhr brick, natural kieselguhr brick perpendicular to grain, "nonpareil" insulating brick, and "nonpareil" high-pressure block. The conductivities increased with rise of temperature up to 1500° F. (815° C.).—H. S. H.

*Vermilion colour [for glazes] from uranium.* C. F. Binns and F. Lytle. *J. Amer. Ceram. Soc.*, 1920, 3, 913—914.

A BRIGHT vermilion colour is given by a glaze corresponding approximately to the mixture: red lead 57%, felspar 20%, zinc oxide 2%, flint 12%, uranium oxide 9%, and fired to cone 04 in closed saggars. The amount of uranium oxide present must not vary greatly. Some difficulty is experienced in protecting the uranium oxide from reducing influences.—H. S. H.

## PATENTS.

*Glass; Means for obtaining* — from a furnace. The Empire Machine Co., Asses. of B. D. Chamberlin. E.P. 138,895, 9.2.20. Conv., 15.11.15.

A GLASS furnace having a suitable orifice through which a stream of glass may flow is provided immediately beneath the orifice with a shearing mechanism through which a cooling medium flows. To prevent "scarring," due to chilling of the glass, the orifice and shearing mechanism are enclosed in a supplemental heating chamber, so that the severed and chilled ends of the glass are immediately reheated. By varying the temperature in the supplemental chamber the rate of flow of the glass and the cross-sectional area of the severed charge are controlled.—H. S. H.

*Glass; Impts. in and apparatus for the manufacture of* — A. Ferguson. E.P. 154,251, 28.5.19.

GLASS-MAKING materials are fed into a trumpet-shaped melting chamber with a dome-shaped top. Gas and air are forced in a tangential direction into the upper part of the melting chamber and by their whirling movement form a vortex into which fall the particles of glass-making materials. The materials react, are driven on to the walls, and drop or drain into the tank furnace placed below, and are refined there by the heat from the gases entering the tank furnace from the melting chamber. Waste gases may be used to preheat the gas and air or the glass-making materials.—H. S. H.

*Glass-annealing leer [lehr].* E. E. Milner and W. J. Lytle, Assrs. to H. L. Dixon Co. U.S.P. 1,361,604, 7.12.20. Appl., 1.8.19.

A LEHR is provided with several groups of heating flues surrounding the major portion of the annealing chamber. In one group of flues the heating medium passes in opposite directions in adjacent flues, whilst in another group the heating medium travels upwards and in the same direction in adjacent flues. The upper portion and sides of the lehr are covered with heat-insulating material of progressively decreasing thickness towards the rear end of the annealing chamber.—A. B. S.

*Basic refractory composition and process of making the same.* H. P. Bassett. U.S.P. 1,360,355, 30.11.20. Appl., 15.10.19.

THE composition consists of a mixture of magnesian limestone, iron, a compound of silicon, and sodium chloride.—A. B. S.



**Kiln; Combined muffle and open tunnel** — P. d'H. Dressler, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. (A) 1,360,625 and (B) 1,360,626, 30.11.20. Appl., (A) 26.1.23; (B) 28.5.19. (A) Renewed 18.5.20.

(A) A TUNNEL kiln has an elongated kiln chamber formed of two sections placed end to end. A muffle extends longitudinally in one section and communicates with the other section at the junction of the two sections. The burning gases and products of combustion flow longitudinally through the interior of the muffle and the interior of the other kiln section. (B) In a tunnel kiln with a kiln chamber formed of two sections as in (A), a combustion chamber, separate from the kiln chamber, extends longitudinally in one of the sections and communicates with the other section at the junction of the two sections. Air and fuel enter the combustion chamber at the end remote from the other section. The products of combustion are drawn from the combustion chamber through the other section.—H. S. H.

**Dryer for clay or ceramic products.** I. M. Justice, Assr. to The Manufacturers' Equipment Co. U.S.P. 1,360,645, 30.11.20. Appl., 3.5.19.

THE dryer comprises drying tunnels with fuel passages extending beneath and means for moving the ware through the tunnels. Air enters through inlets placed at the receiving ends of the drying tunnels and is gradually heated as it passes through the dryer. The fuel tunnels and the drying tunnels communicate with a stack.—H. S. H.

**Enamel coat on steel; Process for producing a single white** — S. Rieser, Assr. to The New England Enameling Co. U.S.P. 1,360,317, 30.11.20. Appl., 21.11.16.

In the production of white enamel on steel the frit is ground with water and clay, and a portion only of the soluble enamelling constituents removed.

—H. S. H.

**Enamels; Process for making** — G. Rupprecht, Assr. to The Chemical Foundation, Inc. U.S.P. 1,360,585, 30.11.20. Appl., 4.6.17.

ENAMELS are made by subjecting the raw materials to the direct action of a flame, the molten substance being supported on an unmolten mass of the same raw materials and prevented from coming into contact with foreign matter.—A. B. S.

**Glass surfaces; Decorating** — K. Wurga. E.P. 148,304, 9.7.20. Conv., 16.8.16.

SEE U.S.P. 1,283,606 of 1918; J., 1919, 76 A.

**Glass; Apparatus employed in manufacture of hollow articles of** — A. Ferguson. E.P. 154,252, 28.5.19.

**Glass; Delivery of molten** — [to moulds]. British Hartford-Fairmont Synd., Ltd. From Hartford-Fairmont Co. E.P. 154,861, 25.6.20.

## IX.—BUILDING MATERIALS.

**Lime plaster; "Popping" (unsoundness) of** — W. E. Emley and C. H. Bacon. J. Amer. Ceram. Soc., 1920, 3, 877—884.

THE "popping" of a lime plaster is caused by grains of a material which hydrates slowly and expands as it hydrates. Such material may be a compound of calcium with silica, alumina, or iron, or an incompletely oxidised salt of iron, and may come from the ash of the fuel or from impurities in the limestone or sand. By rejecting all lumps of lime encrusted with vitreous material, screening

the hydrated lime through a No. 48 sieve, and soaking all lime putty overnight before using it as a plaster, all danger of "popping" is removed. The sand should not contain more than 1% of sodium and magnesium chlorides.—H. S. H.

## PATENTS.

**Exotic woods; Process for reducing or softening the colours of** — P. E. Simon. E.P. 141,052, 30.3.20. Conv., 9.4.18.

THE wood is immersed in acid, preferably nitric acid (sp. gr. 1.04—1.26), then dried rapidly at the maximum temperature which it can endure without charring, and afterwards well rinsed in water. Rosewood veneers are immersed for  $\frac{1}{2}$  hr. in oxalic acid solution at 80° C., rinsed in water at the same temperature, and the moist wood treated as described above. A mixture of hydrochloric and nitric acids of sp. gr. 1.02—1.06 at 30°—40° C. may be used instead of nitric acid if a redder colour is desired. To lessen the visibility of the grain a preliminary immersion for 24 hrs. in 90% alcohol followed by hot rinsing is recommended. Amaranth wood is immersed for 15—30 mins. in water at 60°—80° C. and then in a nitric acid bath. The treated wood may be scraped without change of colour.—A. B. S.

**Glaze on building materials, in particular cement; Method of producing a cold** — K. Friedrich. E.P. 154,236, 27.3.18.

A GLAZE is produced on concrete, stone, brickwork, and the like by applying a mixture of 8 pts. of finely sifted Portland cement, 1 pt. of colouring matter, 10 pts. of water, and 1 pt. of bituminous substance prepared by dissolving 15 pts. of sodium carbonate in 60 pts. of water and adding 20 pts. of asphaltum (freed from oils of b.p. below 270° C.), shaking the mixture to emulsify it, and stirring in 5 pts. of powdered barium peroxide.—A. B. S.

**Shaft furnaces especially applicable to the manufacture of cement.** F. W. Bakema. E.P. 154,510, 8.5.20.

THE lower part of a shaft kiln for burning Portland cement is fitted with a perforated rotating helix which acts as a grate. The helix may be supported axially, or partly axially and partly by attaching its circumference to a rotating drum, to provide a better support.—A. B. S.

**Bricks; Process for the manufacture of light** — from freshly mined kieselguhr. W. Bunte. G.P. 326,116, 21.10.19. Addn. to 324,375.

FRESHLY mined kieselguhr containing up to 50% of moisture is ground together with cheap binding materials, e.g., gypsum or lime, or both, instead of cement. Sand or similar substances may also be added.—A. R. P.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Nickel steels; Cause of the instability of** — and its elimination. C. E. Guillaume. Comptes rend., 1920, 171, 1039—1041.

THE instability of nickel steels is shown to be due to the presence of carbon and the consequent formation of cementite, the slow transformation of which is capable of producing the phenomena observed (cf. J., 1899, 763). The stabilisation of nickel steels may thus be connected with the absence of cementite. It is shown that the addition to nickel steel of a sufficient amount of a metal such as chromium, tungsten, or vanadium, which has an affinity for carbon greater than that of iron, renders such steels far more stable, but a slight residue of

cementite always remains. To arrive at complete stability it seems necessary to add to the alloy a considerable excess of the third metal.—W. G.

[Gold;] *Influence of soluble silica and calcium salts on precipitation* [of — from cyanide solutions]. J. H. Johnson. J. Chem. Met. Soc. S. Africa, 1920, 21, 58—59.

THE presence of colloidal silica in the solutions from sand clarifiers led to the formation of a deposit on the zinc in the precipitation boxes and of a scum at the head and between compartments of the boxes. The deposit, which prevented the precipitation of gold by the zinc, and the scum were found to consist mainly of calcium carbonate and hydrated silica, probably due to excessive alkalinity of the solutions in the circuit.—A. R. P.

*Silver; Recovery of — from manganese-silver ores.* J. A. Carpenter. Eng. and Min. J., 1920, 110, 898—902.

MANGANESE-SILVER ore from which the silver cannot be obtained by any of the ordinary methods owing to its association with refractory minerals, gives good yields if heated to 1000° C. in a current of air with sodium chloride. Silver, copper, lead, and gold chlorides are volatilised and collected in a Cottrell plant with a yield of 95—99%. Alternatively, the ore may be heated with the salt to about 900° C. for 30—45 mins., whereby only small quantities of the precious metals are volatilised, the remainder being leached out by cyanide in the usual way. The extraction is exceedingly rapid and very complete and the cyanide consumption low. The cost of the processes should not be prohibitive; 15—25 galls. of fuel oil is required per ton for the first process and much less for the second, while the consumption of salt varies from 50 to 180 lb. per ton.—A. R. P.

*Copper losses in slags.* F. E. Lathe. Eng. and Min. J., 1920, 110, 1076—1080.

THE amount of oxidised copper present in slags from copper smelting is determined by digesting 2 g. of the slag, ground to pass 200-mesh, with 100 c.c. of a 3% solution of sulphur dioxide and 10—15 c.c. of hydrofluoric acid in a stoppered bottle for several days with occasional agitation. The solution is filtered, the filtrate boiled to expel sulphur dioxide, and the copper precipitated as sulphide and determined electrolytically. The estimation of the sulphide copper in slags by digestion with silver nitrate solution is unreliable, as the reagent dissolves a small part of the oxidised copper and leaves part of the sulphide copper unattacked. Tables are given showing the proportion of copper existing in the two states in blast furnace, reverberatory, and converter slags from several American smelters, and suggestions are given for possible improvements in the technique of slag settling.—A. R. P.

*Roasted blende; Determination of the sulphide sulphur in —.* M. de Kegel. Rev. Chim. Ind., 1920, 29, 285—288. Chem. Zentr., 1920, 91, IV., 659—660.

DISTILLATION of the material with stannous chloride and absorption of the evolved hydrogen sulphide in a solution of zinc or cadmium acetate and iodine in potassium iodide gives unreliable results. The following procedure is therefore recommended: The finely divided sample is heated with a solution of stannous chloride in hydrochloric acid and the evolved hydrogen sulphide driven, by means of a current of carbon dioxide, through a series of three absorption flasks, the first of which contains dilute acetic acid and the other two, cadmium acetate solution. When the reaction is complete, the contents of the latter two flasks are boiled for 10 mins. and allowed to cool for 15 mins. in a current of carbon dioxide. The liquid is then transferred to

a larger flask, boiled to expel carbon dioxide, cooled, diluted largely, a measured volume of standard iodine added, and the excess titrated as usual. Addition of a small quantity of ammonium carbonate when diluting, increases the sensitivity of the end-point. Stoppers of vulcanised rubber must not be used in any part of the distillation apparatus.—A. R. P.

*Lead [in ores]; Determination of — as chromate.* C. W. Simmons, J. R. Gordon, and H. C. Boehmer. Canad. Chem. J., 1920, 4, 139.

THE ore is dissolved in a mixture of hydrochloric and nitric acids, the solution evaporated to a volume of 8 c.c., and ammonia added until a permanent precipitate of hydroxide is obtained; this is dissolved by adding an excess of acetic acid and 3 g. of ammonium acetate, and 10 c.c. of 10% potassium chromate solution is then added. The mixture is kept warm for 5 mins., the precipitated lead chromate collected, dissolved in a mixture of 25 c.c. of concentrated hydrochloric acid and 75 c.c. of water, the solution diluted to 200 c.c., treated with 1 g. of potassium iodide, and the liberated iodine titrated with thiosulphate solution.

—W. P. S.

*Metals; Solubility of — in acids containing formaldehyde.* R. C. Griffin. J. Ind. Eng. Chem., 1920, 12, 1159—1160.

THE solvent action of dilute sulphuric acid (10° B., sp. gr. 1.074) and hydrochloric acid (1.1) on wrought iron, cast iron, and steel is decreased very considerably by the addition of 1% of formaldehyde to the acids, the corrosion loss being diminished 65—98%. The effect is less marked in the case of 10% nitric acid and also in the case of other metals, such as brass, tin, nickel, etc.—W. P. S.

*Hydrogen peroxide; Use of — in metal tinting.* H. Krause. Metall, 1920, 229—230. Chem. Zentr., 1920, 91, IV., 715.

HYDROGEN peroxide is of no use for tinting smooth polished metal surfaces, but for unpolished surfaces, especially those that have been coloured by other reagents, e.g., a strong solution of potassium chlorate containing ammonium nitrate, or, for darker tints, potassium permanganate, concentrated solutions of hydrogen peroxide (perhydrol) are useful for toning down the colours, for which purpose the reagent is applied with a fine brush and develops generally a bright brown colour.—A. R. P.

*Bismuth in ores.* Schoeller and Waterhouse. See XXIII.

#### PATENTS.

*High-speed steel; Treating —.* C. A. Whitmyre, Assr. to General Electric Co. U.S.P. 1,359,851, 23.11.20. Appl., 2.8.19.

THE steel is hardened by heating to 1275°—1300° C., then quenching at 600°—650° C.—J. W. D.

*Steel; Process of improving the machining properties of —.* M. T. Lothrop, Assr. to The Timken Roller Bearing Co. U.S.P. 1,360,551, 30.11.20. Appl., 20.10.19.

ROLLED steel is hot worked at a temperature above 2300° F. (1260° C.) to improve its machining properties.—T. H. Bu.

*Iron and steel; Manufacture of — directly from the ore, and apparatus therefor.* L. P. Basset. U.S.P. 1,360,711, 30.11.20. Appl., 24.10.18.

THE ore is reduced by means of a carbonaceous reducing agent, and the metal melted by a very hot flame producing substantially only carbon monoxide, with exclusion of carbon dioxide and water vapour.—J. S. G. T.

*Steel; Method for manufacturing* — D. R. Knapp. U.S.P. 1,361,689, 7.12.20. Appl., 20.1.20.

AFTER melting and refining a charge of steel, a relatively small portion of the charge is removed and a relatively small portion of cold charge added to the molten bath.—T. H. Bu.

*Alloys of copper and zinc; Electro-deposition of* — S. O. Cowper-Coles. E.P. 154,108, 24.2.20.

CATHODE plates or articles to receive the deposit are placed in an electrolyte consisting of a solution of a salt or compound of one of the metals, and a salt or compound of the other metal is added continuously, the electrolyte being rapidly agitated with propellers. A test-piece of thin metallic ribbon is passed continuously through the electrolyte and is slowly wound up by suitable mechanism in order to facilitate control of the working conditions.

—J. W. D.

*Electric [smelting] furnaces.* A. M. Eriksen. E.P. 154,444, 25.11.19.

IN electrical smelting furnaces provided with resistance bars above the charge, adjustable vents are provided by means of which a slight draught is created through the space above the charge. Deposition of condensed matter in the sleeves in the furnace walls through which the resistance bars pass is thus prevented and freedom of movement of the bars maintained. The charging hopper projects downwards through the heating bar grate, whereby preheating of the charge in the hopper is effected on both sides. The furnace may be mounted on wheels turning about the tilting axis of the furnace.—J. S. G. T.

*Electrolysing a solution of nickel salt; Method for* — C. Heberlein. E.P. 154,471, 8.8.19.

THE process is carried out in a cell of the type described in E.P. 141,766 (J., 1920, 456 A), comprising a series of electrolytic vats, each divided into a number of cathode compartments containing a similar number of stationary cathodes separated by electrolytic diaphragms from insoluble anodes arranged between the cathode compartments, the whole being immersed in a single anode compartment formed by the vat. The catholyte is fed in parallel, though the cathode compartments of one vat after the other, whereby uniform concentration and acidity are maintained in each compartment. The catholyte is prepared by dissolving nickel from ore, matte, etc., by means of acid until an acid liquor of the optimum acidity (0.1–0.4% of free acid) for electrolysis is obtained. A nickel salt solution is used as anolyte and circulates through the anode compartments in series. Circulation of catholyte and anolyte is continued until the acidity of the anolyte is such as to be incompatible with the maintenance of optimum acidity of the catholyte. Thereupon the anolyte is withdrawn and used for preparing fresh nickel salt solution of optimum acidity, whilst the catholyte is returned to the catholyte circulation in admixture with fresh nickel salt solution, so that a catholyte liquor of optimum acidity is obtained.—J. S. G. T.

*Electrolysis [for deposition of metal in non-coherent form].* W. A. Adam, J. Stevenson, A. T. Mahitt, and J. Fieldhouse. E.P. 154,635, 27.2.19.

ELECTROLYTIC deposition of metals in a non-coherent form is effected in an electrolytic cell provided with a rotating drum cathode preferably composed of carbon impregnated with paraffin wax or the like. A cell having a number of cathodes rotating in the direction of flow of the electrolyte may be employed. The carbon anode is of trough form, and a narrow annulus for passage of electrolyte is left between the anode and cathode. The surface of the anode is impregnated with paraffin wax or the like. The

active area of cathode surface can be regulated by means of adjustable rubber pads. The depth of electrolyte is regulated by adjustable weirs, and the deposit is removed from the cathode by means of a shoot or scraper, a wiper or the like serving to propel the deposit along the shoot.—J. S. G. T.

*Alloy for electrical contact-points.* J. A. Williams. U.S.P. 1,358,908, 16.11.20. Appl., 28.10.18.

THE alloy consists of silver with less than 1% of lead.—J. H. L.

*Compound metallic articles; Method of making* — G. A. Mead, Assr. to The Ohio Brass Co. U.S.P. 1,359,719, 23.11.20. Appl., 22.10.19.

AN article of two dissimilar metals is made by coating the fused surface of one of the metals with a dissimilar metal of lower melting point to form a bond, to which the fused second metal is then applied at a temperature higher than the melting point of the coating metal.—J. S. G. T.

*Copper; Method of hardening* — B. Kieffer. U.S.P. 1,359,810, 23.11.20. Appl., 2.2.20.

COPPER is heated to a temperature somewhat below its melting point, finely divided glass is applied to its surface, and it is then cooled rapidly.—J. W. D.

*Alloy of alkaline-earth metals; Production of* — G. J. Kroll. U.S.P. 1,359,813, 23.11.20. Appl., 30.1.20.

A HEATED mixture or alloy of an alkali metal (or magnesium or beryllium) is treated with a molten mixture of chemical compounds containing at least one halogen compound of the alkaline-earth metal to be alloyed.—J. W. D.

*Alloy; Hard lead* — W. A. Cowan, Assr. to United Lead Co. U.S.P. 1,360,269, 30.11.20. Appl., 11.8.19.

A HARD lead alloy contains lead with a small percentage of lithium.—T. H. Bu.

*Alloy; Lead-base* — E. De Campi, Assr. to United Lead Co. U.S.P. 1,360,272, 30.11.20. Appl., 30.7.19.

ALUMINIUM is mixed with a metal or metals capable of alloying with it, and the mixture introduced into alloys composed mainly of lead.—T. H. Bu.

*Alloy; Hard lead* — T. F. Weltstein, Assr. to United Lead Co. U.S.P. 1,360,339, 30.11.20. Appl., 2.10.18. Renewed 28.5.20.

A HARD lead alloy contains at least 97% Pb, the remainder being barium and calcium; the amount of barium is  $1\frac{1}{2}$  to 8 times that of calcium.

—T. H. Bu.

*Alloys; Lead-base — Lead alloy.* G. H. Worrall, Assr. to United Lead Co. U.S.P. (A) 1,360,346 and (S) 1,360,347, 30.11.20. Appl., 11.8.19. Renewed 28.5.20.

HARD lead alloys suitable for bearings with lead as the principal constituent contain in addition (A) 3% or less of one of the alkali metals and 2% or less of mercury, or (S) 2% or less of magnesium and 2% or less of mercury. (Cf. E.P. 141,122 of 1919; J., 1920, 414 A.)—T. H. Bu.

*Hard lead alloy.* G. H. Worrall, Assr. to United Lead Co. U.S.P. 1,360,348, 30.11.20. Appl., 25.8.19. Renewed 28.5.20.

LEAN alloys of predetermined hardness are made by hardening a lead-base alloy containing upwards of 90% Pb by adding a small percentage of mercury.—A. B. S.

**Bismuth; Process of extracting** — J. F. Cullen and M. J. Udy, Assrs. to United States Smelting, Refining, and Mining Co. U.S.P. 1,360,271, 30.11.20. Appl., 19.3.18.

THE ore or other material is leached with brine containing an acid to form a solution of the bismuth salt of the acid, and the bismuth is then precipitated from the solution.—T. H. Bu.

**Smelting; Process of** — J. Lund. U.S.P. 1,360,552, 30.11.20. Appl., 10.12.17.

A "PUFFED" slag, in the pores of which sodium carbonate and petroleum coke have been precipitated, is introduced into a furnace in proximity to the charge, and the material and slag are heated, the latter acting as a flux.—T. H. Bu.

**Alloy**. F. Milliken, Assr. to F. Milliken, S. F. Weaver, and J. M. Repplier. U.S.P. 1,360,773, 30.11.20. Appl., 8.10.19.

THE alloy contains 8–12% Fe, 55–65% Cu, 12–18% Ni, 11–17% Zn, and about  $\frac{1}{2}$ –1% Si.  
—T. H. Bu.

**Metallurgical process**. G. T. Hansen. U.S.P. 1,361,459, 7.12.20. Appl., 10.1.19.

GOLD, silver, and copper are extracted from ores by leaching the ore with a solution of potassium cyanide at ordinary temperatures, collecting and heating the solution to 90° F. (32° C.) or higher, and subjecting the hot solution to the action of a metal, such as zinc, aluminium, etc.—T. H. Bu.

**Electrolytic de-tinning bath for the complete or partial de-tinning of tin-plate**. P. Rocks. G.P. 326,048, 22.1.20.

AN aqueous solution of aluminium chloride is used as the electrolyte. In order to recover the tin from one side only of tinned metal, two sheets are placed together to form the anode in a 3% aluminium chloride solution with a cathode on each side.  
—D. F. T.

**Steel; Process of manufacturing electric** — J. McConnell. E.P. 154,659, 30.7.19.

SEE U.S.P. 1,318,164 of 1919; J., 1919, 908 A.

**Steel; Manufacture of** — W. L. Turner. U.S.P. 1,360,830, 30.11.20. Appl., 7.1.18.

SEE E.P. 123,102 of 1917; J., 1919, 224 A.

**Smelting ores, melting metals, and the like; Combustion processes and apparatus for** — U. A. Garred. E.P. 154,240, 9.8.18.

SEE U.S.P. 1,311,807 of 1919; J., 1919, 685 A.

**Separating magnetic material; Method and apparatus for** — G. Ulrich, Assr. to Chemical Foundation, Inc. U.S.P. 1,360,601, 30.11.20. Appl., 9.2.16. Renewed 19.4.20.

SEE E.P. 100,064 of 1916; J., 1916, 475.

**Ores and other materials; Apparatus for washing and classifying** — R. Haddan. From The Dorr Co. E.P. 154,512, 17.5.20.

**Wire; Process and apparatus for manufacture of — by electro-deposition**. S. O. Cowper-Coles. E.P. 154,833, 18.3.20.

**Slime-pulp thickener**. U.S.P. 1,360,703. See I.

**Alkali-resisting articles**. G.P. 326,032. See I.

**Washing coal etc.** E.P. 153,790. See IIA.

## XI.—ELECTRO-CHEMISTRY.

### PATENTS.

**Electric furnaces**. D. F. Campbell. E.P. 153,951, 14.8.19.

THE furnace electrodes are adapted to be tilted and fed by mechanism supported independently of the furnace body, so that the furnace may be employed either as an arc or resistance type of furnace. The electrodes, when tilted, are brought into contact with carbonaceous material, such as coke, applied to the surface of the charge.—J. S. G. T.

**Electric furnace. Process of treating charge materials in electric furnaces**. C. E. Parsons. U.S.P. (A) 1,359,735 and (S) 1,359,736, 23.11.20. Appl., 19.2.20.

(A) MEANS are provided for feeding the material to be treated in the furnace in an upward direction between the electrodes, for withdrawing it therefrom in a downward direction, for maintaining the interior of the furnace airtight, and for subjecting the material to suction while being treated. (S) Material to be treated is fed to and withdrawn from the furnace as described under (A). The material is made electrically conducting and then when molten is caused to impinge against a sharp edge to prevent sticking.—J. S. G. T.

**Electrical oxidation of gases; Furnaces for** — B. Thomas. U.S.P. (A) 1,359,932 and (S) 1,359,933, 23.11.20. Appl., 18.7.17.

(A) THE furnace electrodes are formed of closely coiled pipe and are placed one within the coils of the other, so as to form an annular arcing chamber. Means are provided for circulating a cooling fluid through the pipes. (S) Two concentric electrodes are spaced so as to form an annular air inlet passage which is provided with inclined vanes to impart a whirling motion about the axis of the electrodes to the incoming air.  
—J. S. G. T.

**Electrolysers; Bi-polar electrode** — G. G. Hephurn, and Mather and Platt, Ltd. E.P. 154,029, 26.9.19.

IN bi-polar electrode electrolysers of the type described in E.P. 12,730 of 1915 (J., 1916, 1120) side and bottom walls or divisions are provided between the electrodes so that the space between the electrodes is open only at the top. Channels for drawing off electrolyte extend from the active face of an electrode and through it to the end face, and an aperture for the same purpose is arranged in the side wall either near the bottom of the cell, or near the top, with an intermediate side wall extending over the aperture nearly to the bottom of the cell.  
—J. S. G. T.

**Electrolytic cell [; Anode for —]**. E. A. Le Sueur. U.S.P. 1,359,716, 23.11.20. Appl., 13.5.18.

AN anode for an electrolytic cell consists of a carbon plate extending laterally and downwards from a carbon lead, with another carbon plate of smaller width extending laterally from the lead above the first plate and substantially in the same plane.  
—J. S. G. T.

**Electrolytic cell**. G. K. Thomas. U.S.P. 1,360,055, 23.11.20. Appl., 17.5.20.

ONE of the electrodes of the cell is enclosed within an envelope provided with apertures through which alone the electrolyte is able to come into contact with the enclosed electrode.—J. S. G. T.

**Electrolytic apparatus. Water-feed attachment for electrolytic cells. Electrode.** I. H. Levin. U.S.P. (a) 1,360,541, (b) 1,360,543, (c) 1,360,544, and (n) 1,360,545, 30.11.20. Appl., (a) 25.10.15, (b, c, n) 21.4.19. All renewed 23.4.20.

(A) An electrolytic apparatus of the filter-press type comprises a number of electrodes separated by diaphragms and provided with openings lined with insulating material forming independent feed ducts for electrolyte. (b) A chamber for feed water is provided with passages discharging within the electrolytic cell. Baffles are arranged within the chamber, above and near the ends of the passages, and a perforated shield is placed above the baffles. Gas derived from bubbles bursting in the chamber, passes through the shield, and the liquid envelopes of the bubbles are returned to the chamber. (c) The cell casing is provided with an opening, the walls of which converge towards the inside of the cell, and into which a tapered resilient body of insulating material, provided with an opening through which the electrode terminal passes, can be forced. (n) An electrode comprises two sheet metal plates having oppositely disposed depressions near the top, and separated by a flat metal terminal bar of about the same width as the depressions. The plates and bar are mechanically and electrically connected by a weld joint, whereby electrolyte is excluded from between the plates and the bar.

—J. S. G. T.

**Electrolytic apparatus.** I. H. Levin, Assr. to Electrolytic Oxy-Hydrogen Laboratories, Inc. U.S.P. 1,360,542, 30.11.20. Appl., 25.5.17. Renewed 23.4.20.

An electrolytic tank is divided into a number of chambers by a partition in electrical connexion with the lower portion of the tank. The partition is provided with an opening. An inner electrode enclosed by a diaphragm is contained within each chamber. The gas-tight cover of the tank carries a number of inverted troughs projecting to a point below the normal level of electrolyte, each inverted trough being associated with an elongated trough the sides of which merge into the sides of the respective inverted troughs. The tops of the elongated troughs are partly open, but are closed where the walls of the troughs merge into one another. Means are provided whereby the electrolyte and water for diluting the electrolyte may flow from respective chambers into an elongated trough on either side of the wall of the inverted trough. A feed cup is provided with ducts leading to one of the chambers formed by the partition and a gas offtake system communicates with all the inverted troughs.

—J. S. G. T.

See also pages (A) 45, *Manganese chloride* (U.S.P. 1,359,640); *Permanganate* (U.S.P. 1,360,700); *Oxides* (U.S.P. 1,361,041); 49, *Electric furnaces* (E.P. 154,444); *Electrolysing nickel solution* (E.P. 154,471); *Electrolysis* (E.P. 154,635); *Alloy for electrical contact points* (U.S.P. 1,358,908).

## XII.—FATS; OILS; WAXES.

**Soya bean oil; Direct identification of —.** C. A. Newhall. J. Ind. Eng. Chem., 1920, 12, 1174—1175.

FIVE c.c. of the oil is mixed with a few drops of gum arabic solution and 5 c.c. of 2% uraninm acetate or nitrate solution, 5 c.c. of chloroform is added, and the mixture is shaken thoroughly. Crude and refined soya bean oils yield a characteristic yellow emulsion, but other oils, with the exception of linseed oil, give a white or very slightly coloured emulsion; linseed oil gives a light brownish

colour. The test will detect the presence of 5% of soya bean oil in earthenut (arachis), cottonseed, sesame, rape, and coconut oils.—W. P. S.

**Prickly pear seed oil.** S. Lomanitz. J. Ind. Eng. Chem., 1920, 12, 1175.

PRICKLY pear seeds yield about 11% of a semi-drying oil having the following characters:—Sp. gr. at 15.5°/15.5° C., 0.9294;  $n_D^{20}$  = 1.4676; acid value, 3.09; saponif. value, 189.5; iodine value, 116.3; Reichert-Meißl value, 2.8; insoluble fatty acids, 93.81%.—W. P. S.

**Viscosity at high pressures. Compressibility of lubricating oils.** Hyde. See 11A.

**Viscosity at high pressure.** Parsons. **Viscosity at any pressure.** Boys. **Cup and ball viscosimeter.** Thomsen. See 11A.

**Oil emulsions.** See 11A.

**Hydrolysis of cottonseed hulls.** Sherrard and Blanco. See XVIII.

## PATENTS.

**Fish oils; Deodorisation and other purification of —.** De Nordiske Fabrikker De-nor-fa Aktieselskab. E.P. 140,372, 13.1.20. Conv., 18.3.19.

WHALE or other fish oil, after treatment with a solution of sodium hydroxide or sulphuric acid, is hydrogenated in an autoclave for 4–6 hrs. at 150°–200° C. in the presence of a small quantity of nickel catalyst until the iodine value is reduced from about 125 to about 100. The hydrogenated oil, after filtration, is slightly polymerised by heating to 260°–280° C. for 3–4 hrs. in a closed vessel through which hydrogen is passed, and, after cooling to 80°–100° C., is finally deodorised by treatment *in vacuo* with steam superheated to 250°–300° C.

—L. A. C.

See also pages (A) 52, *Solidifying tung oil* (E.P. 153,942); 53, *Fat from organic matter* (E.P. 137,843); 55, *Glycerin substitute* (G.P. 325,647); 57, *Glycerol* (E.P. 138,328); 59, *Waste organic substances* (U.S.P. 1,359,085–6); 62, *Perhydronaphthylphenylmethane-o-carboxylic acids* (G.P. 325,714).

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Paints; Detection of oils other than linseed in — by means of the hexabromide value of the fatty acids.** H. Bailey and W. D. Baldisien. J. Ind. Eng. Chem., 1920, 12, 1189–1194.

THE hexabromide value of pure linseed oil is 42, of soya bean oil 6, and of fish oils 0; the latter, however, yield ether-insoluble octobromides which can be separated from the hexabromides of the other oils by taking advantage of their insolubility in warm chloroform. The method adopted for brominating the fatty acids consists, briefly, in dissolving 1 g. of the fatty acids in 25 c.c. of ether and 2 c.c. of glacial acetic acid, cooling the solution to –10° C., and adding about 25 c.c. of a mixture of 20 c.c. of bromine and 80 c.c. of glacial acetic acid.—W. P. S.

**Varnishes with furfural.** C. Coffignier. Bull. Soc. Chim., 1920, 27, 865–867.

FURFURAL, introduced into varnishes with resinsates (cf. F.P. 494,300 of 1918), plays exactly the same part as does turpentine. Like this latter solvent it evaporates during the drying of the layer of varnish. Its rôle is thus simply that of a volatile solvent.—W. G.

## PATENTS.

*Solvents; Process for the recovery of — [from surface coatings of dopes, etc.].* E. I. Du Pont de Nemours and Co., Asses. of T. Baker. E.P. 130,962, 5.5.19. Conv., 8.8.18.

THE object in process of being coated with a composition containing an alcohol is supported adjacent to a vaporising chamber, and gradually advanced through a restricted, adjustable opening in the latter as successive portions are coated. The vapour-laden air is withdrawn from the chamber through a cooler and thence into an absorbing agent consisting essentially of wood tar oil, from which the alcohol is recovered by distillation.—W. E. F. P.

*Oleaginous substances [tung oil]; Process of producing solidification of —.* B. Scobel. E.P. 153,942, 5.8.19.

A SOLID material suitable for making tiles for flooring, or other purposes is prepared by agitating a solution of a hydrated or anhydrous metallic halide, e.g., ferric chloride, in an anhydrous solvent, such as acetone, with tung oil; the mixture is allowed to stand or subjected to vacuum to remove trapped air, and is subsequently heated in moulds to 80°–100° C. to cause solidification. Application of pressure during this stage increases the firmness and density of the product. Fillers such as silica or insoluble silicates, Prussian blue, graphite, iron or chromium oxides, wood or cork flour, rosin, or gums may be added to the oil before treatment.—L. A. C.

*White pigment.* P. Farup, Assr. to Titan Co. A/S. U.S.P. 1,360,737, 30.11.20. Appl., 10.3.19.

A TITANIUM hydroxide is intimately mixed with a small amount of phosphoric acid, or a titanium oxygen compound containing iron is treated with sulphuric and phosphoric acids, the product heated with sodium chloride, and the resulting mass lixiviated.—C. I.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Caoutchoucs [; Artificial —], particularly two dimethylcaoutchoucs [methyl-rubbers].* H. Pohle. Kolloidchem. Beihefte, 1920, 13, 1–60. Chem. Zentr., 1920, 91, IV., 683–684.

AN extensive comparison is made of the properties of two dimethylbutadiene rubbers, covering the effect of colloidal coagulation and dispersion, solubility, oxidisability, and absorption of benzene vapour. A coagulation structure has no lasting influence on the properties of the rubber, and the solubility relations, although affected by resins, are unaffected by protein matter. Methyl-rubber containing an added organic solvent in place of resins shows improved elasticity and resistance to atmospheric action, but, on account of the volatile character of the organic liquid, tends to develop porosity on vulcanisation. Three "ideal" conditions are recognisable in rubber, the absorbent-insoluble, absorbent-soluble, and soluble-non-absorbent; good natural rubber exhibits a combination of the first and second, whereas synthetic rubbers generally are distinguished by the additional presence of the third condition. In the presence of the third condition, rubber shows a greatly increased absorbent power for benzene vapour. According to this view the mechanical working of rubber does not effect any true depolymerisation.—D. F. T.

*Rubber articles; Microsections cut from vulcanised —.* H. A. Depew and I. R. Rnby. J. Ind. Eng. Chem., 1920, 12, 1156–1159.

MICROSCOPICAL examination of sections of coloured rubber articles affords valuable information as to the dispersion of the pigment; the examination also

aids in the identification of the compounding ingredients, such as reclaimed rubber, fibre, pigment, etc. The presence of flocculated pigment possibly accounts for the heating up observed in the case of tire treads heavily compounded with carbon black.—W. P. S.

*Rubber compounds; Ageing of certain —.* I. R. Ruby and H. A. Depew. J. Ind. Eng. Chem., 1920, 12, 1155–1156.

VARIOUS fast-curing motor tyre tread compounds were subjected to an accelerated ageing test in an air-bath at 70° C. The results showed that a compound containing lithopone aged somewhat less satisfactorily than one containing zinc oxide; a compound containing carbon black was much inferior to the others as regards tensile strength and elongation after ageing.—W. P. S.

*Antimony in rubber goods; Determination of —.* S. Collier, M. Levin, and J. A. Scherrer. India-rubber J., 1920, 60, 1297–1298.

THE sample (0.5 g.) is extracted with acetone and, if mineral oil or "substitute" is present, further with chloroform until the extract is no longer coloured; after drying in a vacuum, the material is heated with cymene (25 c.c.) at 130°–140° C. in a 300 c.c. flask until the rubber has completely dissolved; the cooled liquid is diluted with 250 c.c. of light petroleum spirit (max. h. p. 45° C.) and the mixture left overnight, being then decanted through a Gooch crucible; after washing ten times with petroleum spirit the residue is dried and shaken with hydrochloric acid (30 c.c.) until the antimony sulphide has passed into solution; the solution is filtered slowly through the dried Gooch crucible and, after dilution, the antimony is precipitated with hydrogen sulphide; the antimony is then estimated, e.g., by heating with sulphuric acid (12–15 c.c.) and potassium sulphate (5 g.) in a Kjeldahl flask until a colourless solution is obtained, diluting to about 100 c.c. with water, adding hydrochloric acid (20 c.c.) and sodium sulphite (1–2 g.), boiling to expel all sulphur dioxide, and titrating with N/10 permanganate.—D. F. T.

## PATENTS.

*Rubber substitute and process for producing same.* H. H. Haseltine and M. Gregory, Assrs. to Western Rubber Co. U.S.P. 1,360,744, 30.11.20. Appl., 16.3.18. (Cf. E.P. 123,114 of 1917; J., 1919, 227 A.)

A MIXTURE of fixed oils and sulphur, free from air and moisture, is heated under pressure for a sufficient period to complete the reaction.—D. F. T.

*Rubber; Treatment of raw —.* J. V. Worthington and A. W. T. Hyde, Assrs. to Dunlop Rubber Co., Ltd. U.S.P. 1,360,486, 30.11.20. Appl., 17.5.20.

SEE E.P. 150,043 of 1919; J., 1920, 698 A.

## XV.—LEATHER; BONE; HORN; GLUE.

*Hide-tannin compound; Nature of the — and its bearing on tannin analysis.* J. A. Wilson and E. J. Kern. J. Ind. Eng. Chem., 1920, 12, 1149–1152.

TANNIN once combined with hide substance cannot be removed to an appreciable extent by any amount of washing that would be practicable. Chemical changes are produced in a tan liquor when this is boiled or evaporated, certain non-tannins being converted into substances capable of tanning; such changes can be followed quantitatively by a method described recently by the authors (J., 1920, 522 A), and this method can also be used to study the ageing of leather.—W. P. S.



*Chrome tanning; One-bath — and determination of the tanning strength by means of the agglutination method.* E. Stiasny. *Collegium*, 1920, 479—485.

A CRITICISM of Griliches' work (J., 1920, 633 A). Chromium salts do not split up into a basic portion and a chromosulphuric acid (*cf.* Z. physik. Chem., 1904, 47, 29), but into a basic chromium salt and free sulphuric acid, both of which are present in a chrome tanning liquor. In the early stages of the chrome tanning process there is a preponderant absorption of acid, and in the later stages of the tanning an increasing and finally a preponderant absorption of the basic portion. Objection is taken to Griliches' use of pickled pelt in his experiments. Basicity should be expressed in terms of metal ions and acid ions. Kobert (J., 1917, 297) proposed to determine tannin strength by the coagulating power of different solutions on red blood corpuscles, but the numbers he gave as representing the power of various tannin solutions to coagulate completely red blood corpuscles in no way correspond with the actual values of the tannins in practice, and hence Kobert's deduction that green and violet chromium salt solutions are equally effective is not conclusive. —D. W.

*Sulphuric acid in leather; Behaviour and determination of free —.* W. Moeller. *Collegium*, 1920, 465—470.

A REPLY to Immerhoiser (J., 1920, 731 A), in which it is maintained that the other extraction method of that author (J., 1919, 227 A) is entirely unsuited to the estimation of free sulphuric acid in leather. —D. W.

*Leather; Determination of hydrochloric acid and neutral chlorides in —.* A. W. Thomas and A. Frieden. *J. Ind. Eng. Chem.*, 1920, 12, 1186—1188.

ONE g. of leather is mixed with 200 c.c. of  $M/10$   $\text{NaH}_2\text{PO}_4$  solution and heated at  $100^\circ\text{C}$ . for 2 hrs.; after cooling, the solution is diluted to 250 c.c., filtered, 200 c.c. of the filtrate is acidified with nitric acid, heated, silver nitrate solution and a further quantity of 20 c.c. of concentrated nitric acid are added, the mixture evaporated in the dark to about 50 c.c. (to oxidise and dissolve precipitated organic matter), diluted to 200 c.c., and the silver chloride collected and weighed. The total amount of chloride in the leather is calculated from the weight obtained. A second portion of 1 g. of the sample is then digested with 200 c.c. of 95% alcohol at  $75^\circ\text{C}$ . for 2 hrs., cooled, diluted with alcohol to 250 c.c., and the mixture filtered; 200 c.c. of the filtrate is rendered alkaline with sodium hydroxide, evaporated to remove alcohol, the residual solution treated with nitric acid and silver nitrate, and the silver chloride collected as described above, and weighed. This weight is a measure of the neutral chlorides present, the difference between the two determinations giving the quantity of acid chlorides. —W. P. S.

*Casein; Technical —. I.* W. M. Clark. *J. Ind. Eng. Chem.*, 1920, 12, 1162—1163.

A DESCRIPTION of the measures adopted by the U.S. Government to ensure a supply of casein for the manufacture of waterproof glue used in aeroplanes. A part of the work done by the Dairy Division of the Department of Agriculture is reported in the following abstracts. —W. P. S.

*Casein; Technical —. II. Grain-curd casein.* W. M. Clark, H. F. Zoller, A. O. Dahlberg, and A. C. Weimar. *J. Ind. Eng. Chem.*, 1920, 12, 1163—1167.

A HIGH-GRADE casein may be obtained by heating skimmed milk as free as possible from fat to  $93^\circ\text{F}$ .

( $34^\circ\text{C}$ .) and adding dilute hydrochloric acid (1:8 by wt.) until coagulation just commences; after the curd has settled, about one-half of the whey is drawn off, the curd is broken up thoroughly, and more of the acid is added until a portion of the whey when tested with methyl-red solution indicates an apparent acidity of  $pH=4.8$  to 4.6. The remainder of the whey is then drawn off, the casein washed with water containing a small quantity of hydrochloric acid ( $pH=4.8$ ), collected on a draining cloth, washed further, pressed, ground, and dried at  $125^\circ\text{F}$ . ( $52^\circ\text{C}$ .) —W. P. S.

*Casein; Technical —. III. Methods of analysis.* R. H. Shaw. *J. Ind. Eng. Chem.*, 1920, 12, 1168—1170.

Moisture is determined by drying at  $100^\circ\text{C}$ . under reduced pressure, and ash by treating 3 g. of the casein with 5 c.c. of calcium acetate solution, drying the mixture and igniting it at a low red heat; the amount of  $\text{CaO}$  resulting from the added calcium acetate is deducted from the weight obtained. Sugar is determined by shaking 10 g. of the casein for 4 hrs. with 250 c.c. of 50% alcohol, allowing the mixture to settle, and using 100 c.c. of the clear solution for the determination. Phosphorus is determined by the official (American) method for the determination of phosphorus in fertilisers, whilst calcium may be determined either in the ash or after the casein has been digested with *aqua regia*. —W. P. S.

*Casein; Technical —. IV. Standardisation of the borax solubility test for commercial caseins.* H. F. Zoller. *J. Ind. Eng. Chem.*, 1920, 12, 1171—1173.

THE viscosity of casein in borax solution is at a maximum at a hydrogen ion concentration of  $pH=8.15$ , whilst at  $pH=8.99$  to 9.1 the viscosity is lower, but constant owing to the buffer effect of borax in this region. The concentration of casein chosen for the improved casein-borax test is about 12% of true casein in  $M/5$  sodium borate solution. The differences in the physical structure of caseins have a greater influence on their viscosity than have the normal contaminating substances present in commercial caseins; high-temperature caseins always exhibit a comparatively great initial viscosity. —W. P. S.

#### PATENTS.

*Skins; Method of puering or bating —.* J. T. Wood. E.P. 154,103, 4.2.20.

THE skins are puered or bated on the grain side only by painting that side with an enzyme paste. —L. A. C.

*Organic matter, more particularly animal bodies; Apparatus for boiling and drying — [for recovering glue and fat].* K. Niessen. E.P. 137,843, 13.1.20. Conv., 2.1.17. (*cf.* G.P. 317,818, 318,542, and 319,335-6; J., 1920, 379 A, 498 A.)

IN an apparatus for the recovery of glue and fat from animal bodies an elbow pipe is fitted through the wall of the boiling tank at about the surface of the liquid; this pipe may be rotated by means of an external handle so that the fat and glue water, or fat alone, may be discharged from the chamber. —W. P. S.

*Casein compounds; Process of producing hardened —.* H. P. Bassett. U.S.P. 1,360,356, 30.11.20. Appl., 25.2.20.

CASEIN is precipitated in the presence of a hardening agent, dried until it contains 18—20% of moisture, and then moulded under the action of heat and pressure. —A. B. S.



## XVI.—SOILS; FERTILISERS.

*Soil reaction; Absorption of phosphoric acid and —.* M. Wrangell. Landwk. Vers.-Stat., 1920, 96, 209—262.

SEVERAL series of experiments were carried out on the absorption of phosphate by maize and mustard plants in soils to which the other necessary manurial constituents were added, and in which the reaction was kept either neutral, alkaline, or acid with citric acid or sulphuric acid. The effect of the addition of calcium carbonate to these soils was also observed. The crops were gathered and analysed. The results showed that maize could utilise difficultly soluble phosphates in an acid soil only, and the addition of lime reduced the amount of phosphate absorbed. Mustard, on the other hand, was able to absorb the same phosphates in more alkaline solution. The absorption of lime and phosphate by maize was in the ratio of 3:1, and by mustard 15:1. This ratio affords an indication of the capability of the plant to utilise phosphate and to produce a change of reaction in neutral soils. The solution of the plant nutrients in the soil appeared to depend on the differential absorption of the cations and anions, and on the change in reaction induced thereby. The cations were preferentially absorbed in the early stages of growth, thus rendering the reaction of the soil favourable for the subsequent absorption of phosphate. These processes were accelerated by the addition of basic and acid solutions. The reaction of the soil was most easily regulated by the use of nitrogenous manures, especially ammonium salts and nitrates. Free citric acid was decomposed in a few days, and the carbon dioxide formed appeared to have a considerable influence on other decomposition processes in the soil.

—J. H. J.

*Moisture in field samples of soil; Determination of —.* H. A. Noyes and J. F. Trost. J. Assoc. Off. Agric. Chem., 1920, 4, 95—97.

In determining moisture in field samples of soil less than 10 g. should never be taken if accurate results are to be obtained.—W. G.

*Soil phosphorus; Technique of determination of —.* H. A. Noyes. J. Assoc. Off. Agric. Chem., 1920, 4, 93—94.

THE prepared air-dry soil (10 g.) is digested by the usual Kjeldahl method for total nitrogen, using 0.7 g. of mercuric oxide, and adding 0.5 g. of sodium or potassium nitrate to complete the oxidation. When the liquid is partially cooled, 200 c.c. of water is added, the mixture made up to 250 c.c., filtered, and 25 c.c. of the filtrate is boiled with 15 g. of ammonium nitrate and then treated with 30 c.c. of ammonium molybdate solution, and the phosphorus determined as usual.—W. G.

*Moisture determination [in fertilisers]; New method for —.* G. F. Lipscomb and W. D. Hutchins. J. Assoc. Off. Agric. Chem., 1920, 4, 55—57.

ONE gram of the material is weighed into a small cup which is then lowered into a thick-walled tube having its lower end heated in a steam-jacket. This tube is fitted with a hollow ground-in stopper and has a side tube connected with a pump by means of which the tube is evacuated. The hollow stopper is filled with a mixture of solid carbon dioxide and ether. After being in the apparatus for 5 mins., the cup is removed, cooled in a desiccator, and weighed. This process is repeated until a constant weight is obtained, and as a rule five heatings of 5 mins. suffice. Owing to the short period of heating and the material being in a vacuum there is little risk of decomposition in materials such as cottonseed meal, fish scrap, or other organic substances.—W. G.

*Moisture determinations in fertiliser materials; Double —.* J. O. Clarke. J. Assoc. Off. Agric. Chem., 1920, 4, 57—59.

EMPHASIS is laid on the necessity for double moisture determinations, i.e., both before and after preparing the sample for analysis by grinding, so that percentages may be calculated back to the original sample. These double determinations are particularly necessary in fertilisers where the moisture is above 10% and the percentage of the active constituent is fairly high.—W. G.

*Phosphoric acid in precipitated phosphate; Effect of mass and degree of fineness on the percentage of available —.* H. D. Haskins. J. Assoc. Off. Agric. Chem., 1920, 4, 64—66.

THE precipitated phosphate examined was prepared by neutralising with slaked lime the phosphoric acid obtained in the manufacture of glue. The available phosphoric acid was estimated with ammonium citrate solution neutral to cochineal and to litmus respectively, and a somewhat higher result was as a rule obtained with the second solution where 2 g. samples were used. The availability was appreciably higher when the material was ground to pass a 100-mesh sieve than when ground to pass only a 1-mesh sieve. Further grinding did not increase the availability to any great extent. With materials containing such a high percentage (nearly 40%) of available phosphoric acid it would appear desirable to use only 1 g. of the material instead of 2 g. for the estimation.—W. G.

*Ammoniacal nitrogen in complex manures containing calcium cyanamide and ammonium salts; Determination of —.* J. Froidevaux and H. Vandenberghe. Chim. et Ind., 1920, 4, 612—616.

AMMONIACAL nitrogen cannot be estimated in the presence of calcium cyanamide by distillation with magnesia, as, under such conditions, the cyanamide loses some of its nitrogen as ammonia. The best method is to bubble air through an ice-cold solution of the mixture to which an excess of 40% sodium hydroxide solution has been added, collecting the ammonia in standard acid. If the ammonia evolved is estimated at hourly intervals the curve plotted consists of two straight lines, almost at right angles, joined by a slight curve at a point corresponding to about five hours' distillation. If the two straight lines are continued the point at which they intersect will correspond, within the limits of error, to the nitrogen present in the original mixture as ammonium salts, the second portion of the curve representing the loss of nitrogen from the cyanamide.—W. G.

*Potash in wood ashes etc.* Haskins. See VII.

## PATENTS.

*Fertilisers and method of manufacture.* W. H. Hyatt and E. N. Fellowes. E.P. 153,434, 26.9.19.

NITRE-CAKE in as dry a state as possible is mixed with rather more chalk than is necessary to neutralise the acidity of the cake, and the mixture is ground to a fine powder. The product contains calcium carbonate about 13%, calcium sulphate 7.6%, sodium sulphate 47.6%.—J. H. J.

*Fertiliser and insecticide.* W. B. Baker and M. C. Shepherd. E.P. 154,057, 31.10.19.

NORTH SOMERSETSHIRE granite dust is intimately mixed with freshly slaked lime and charcoal or soot, suitable proportions being 50, 35, and 21 lb. respectively, and the mixture is allowed to stand for a few days before use. The product has both fertilising and insecticidal properties. Analysis of a sample gave: moisture, 1%; phosphoric acid, 0.96%; nitrogen, 1.4%; and potassium, 0.22%.

—W. J. W.

*Sulphur composition. Phosphate composition.* J. G. Lipman. U.S.P. (A) 1,361,596 and (B) 1,361,597, 7.12.20. Appl., 10.4. and 26.8.20.

(A) A DRY composition containing sulphur and latent sulphofying bacteria. (B) A dry composition containing dry pulverised phosphatic material and a dry culture of sulphofying bacteria.—A. B. S.

*Phosphate.* U.S.P. 1,360,248. See VII.

## XVII.—SUGARS; STARCHES; GUMS.

[*Sugar*] juice; *Entrainment and decomposition of — during boiling in evaporators as an explanation of unknown losses.* A. Schweizer. Arch. Suikerind. Nederl.-Indië, 1919, 27, 2010—2013. Int. Sugar J., 1920, 22, 706—707.

A CONSIDERABLE loss of sucrose has been observed during the evaporation of juice in certain factories in Java, and was considered not to be due to entrainment, since negative results were obtained on examining the condenser water by means of  $\alpha$ -naphthol. It is suggested that during evaporation the juice carried forward in the form of spray is "atomised" as the result of the high velocity of the steam, and that in this state decomposition occurs, not only to the stago of invert sugar (*cf.* Abelous and Aloy, J., 1919, 475 A), but even to the production of carbon dioxide and water.—J. P. O.

[*Sugar*] juice; *Entrainment and decomposition of — during boiling in evaporators as an explanation of unknown losses.* F. G. E. Olsen. Arch. Suikerind. Nederl.-Indië, 1920, 27, 2161. Int. Sugar J., 1920, 22, 707—708.

In order to examine Schweizer's theory (*cf. supra*), steam was drawn off at two points in the multiple effect evaporator, *viz.*, just before the juice-separator, and between the juice-separator and the condenser. Examination of the water obtained on condensing the withdrawn steam showed the presence of much sucrose at the first point with a little invert sugar, but less sucrose and more invert sugar at the second, while the condenser water appeared to be free from sucrose. It is, therefore, concluded that in the vapour lines of the evaporator there was a certain amount of entrainment of sucrose in the form of juice spray, and that this sucrose was first inverted and later totally destroyed.—J. P. O.

[*Sugar*] juice; *Entrainment and decomposition of — during boiling in evaporators as an explanation of unknown losses.* J. S. de Haan. Arch. Suikerind. Nederl.-Indië, 1920, 28, 223—271.

Loss of sucrose may occur due to the passing of juice spray from the evaporators into the condenser waters, and yet the latter may indicate negative results on being tested according to the general procedure with  $\alpha$ -naphthol (*cf. supra*), owing to the fact (proved by means of a laboratory apparatus) that the two liquids do not mix together under the conditions prevailing in practice. In the case of molasses spray at 85° Brix the drops reached the bottom of the column of the Torricellian condenser almost unchanged. By installing an efficient form of spray-catcher (which separated the particles of juice by centrifugal force), it was possible in one of the factories to reduce the loss during evaporation from 3.12 to 0.78% of the sucrose in the cane while in two others the improvement was almost equally satisfactory.—J. P. O.

*Glucose [dextrose] and starch; Determination of — by the alkaline potassium permanganate method.* F. A. Quisumbing. Philippine J. Sci., 1920, 16, 581—601.

25 c.c. of the sugar solution (*e.g.*, hydrolysed starch

solution) is boiled with 50 c.c. of  $N/10$ -potassium permanganate solution and 25 c.c. of 0.4  $N$  sodium carbonate, under strictly standardised conditions. The excess of permanganate is determined by means of standard oxalic acid solution, and the quantity of dextrose found by reference to a table. Applied to commercial starch, after hydrolysis by acid or diastase, the method gave results agreeing with those of Munson and Walker's method (J., 1906, 656). Satisfactory agreement was obtained also in the determination of starch in flours, after hydrolysis by saliva or diastase, but after hydrolysis by acid the method gave results about 10% higher than those obtained by Munson and Walker's method.

—J. H. L.

*Starchy materials; Contrary action of soluble chlorides and sulphates on —.* H. Courtonne. Comptes rend., 1921, 171, 1168—1170.

In the presence of solutions of certain chlorides, at the ordinary temperature, starch is slowly converted into starch paste, and this in turn is converted into soluble starch if the mixture is heated to 115° C. The ease with which these changes take place depends on the solubility of the chloride and the concentration of the solution. A saturated solution of magnesium chloride is the most effective. Cold solutions of potassium, sodium, and ammonium chlorides have no such effect, while with hot solutions the action is very slight. Sulphates, on the other hand, retard these changes. In a saturated solution of magnesium sulphate starch grains show no signs of swelling even when heated to 115° C. in a closed vessel and there is no formation of starch paste.—W. G.

*Hydrolysis of sugar-cane fibre.* Sherrard and Blanco. See XVIII.

### PATENTS.

*Adhesives [gum substitutes]; Manufacture of — [and of glycerin substitute].* H. Wallasch. G.P. 325,647, 22.5.19. Addn. to 323,665 (J., 1920, 759 A).

THE formaldehyde and dicyanodiamide can be made to react in the absence of any condensation agent or with other acidic or dehydrating substances than sulphuric acid; the product at a stage before it becomes adhesive is of value as a substitute for glycerin.—D. F. T.

*Decolorising carbons.* U.S.P. 1,359,094. See IIb.

*Fermentable sugars.* U.S.P. 1,358,898. See XVIII.

## XVIII.—FERMENTATION INDUSTRIES.

*Enzymes. IV. Emulsin, cytoase, ereptase, and urease in germinated barley.* D. Maestrini. Atti R. Accad. Lincei, 1920, [v.], 29, ii., 164—166. (*cf.* J., 1920, 244 A, 309 A.)

WHEN treated with 0.3% acetic acid solution, germinated barley yields an extract capable of hydrolysing amygdalin, the emulsin being present in the suspension of the meal as well as in the filtered extract. No cytoase, ereptase, or urease could be detected in germinated barley.—T. H. P.

*Enzymes; Quantitative action of —.* G. Tammann and O. Svanberg. Z. physiol. Chem., 1920, 111, 49—67.

THE ideal action of an enzyme, which the action of emulsin on salicin resembles, is discussed mathematically.—S. S. Z.

*Maltase; Determination of — in yeast. II. R.* Willstätter and W. Steibelt. Z. physiol. Chem., 1920, 111, 157—170.

THE fresh yeast is triturated with ethyl acetate until it liquefies. Water is then added and the mix-

ture is neutralised with ammonia. The yeast suspension is made up to a definite volume with a known maltose solution and a huffer mixture; aliquot portions are withdrawn at two different intervals, the degree of hydrolysis estimated, and the "time value" calculated. The few experiments carried out by this method show that most of the maltase of the yeast is extracted in this way. The method is also suitable for the comparison of the hydrolysis of sucrose and maltose by certain yeasts. —S. S. Z.

*Saccharase [invertase]; Susceptibility of — to heat.* H. von Euler and I. Laurin. *Z. physiol. Chem.*, 1919, 108, 64—115.

A compilation of data in connexion with the inactivation of invertase by heat under various conditions.—S. S. Z.

*Fermentation by a top yeast; Susceptibility of — to the hydrogen ion concentration.* H. von Euler and S. Heintze. *Z. physiol. Chem.*, 1919, 108, 165—188.

EXPERIMENTS with a strain of a top yeast (S B II.), from the Stockholm Södra lästfabrik, showed that the influence of acidity on the rate of fermentation was dependent on the nitrogen content of the medium, and that the optimum hydrogen ion concentration was about  $pH=5$ . In the case of weakly dissociated organic acids, the quantity of the undissociated acid was great in relation to the concentration of the ions and it also influenced the course of fermentation.—S. S. Z.

*Alcoholic fermentation. VIII. Influence of zinc chloride on the alcoholic fermentation of living and killed yeast.* S. Kostytschew and L. Frey. *Z. physiol. Chem.*, 1920, 111, 126—131.

Zinc chloride causes the formation of acetaldehyde in fermentation by hefeanol and dried yeast but not by living yeast. Apart from the aldehyde the sugar is mainly converted into an unknown compound in the presence of zinc chloride. This production of acetaldehyde only occurs when sugar is present. In the case of autofermentation at room temperature only traces of acetaldehyde can be detected. —S. S. Z.

*Alcoholic fermentation. IX. Influence of cadmium and zinc salts on yeast enzymes.* S. Kostytschew and S. Sukkova. *Z. physiol. Chem.*, 1920, 111, 132—140.

Cadmium salts alter to a very marked extent the normal course of fermentation. Most of the sugar is converted into products of unknown composition, and acetaldehyde is produced at the same time. Proteolysis and, to a greater extent, reduction are inhibited by dilute solutions of cadmium salts, and the reduction of acetaldehyde to alcohol is entirely stopped by small quantities of these salts. Acetaldehyde is produced not only in the presence of oxygen but also under anaërobic conditions. Cadmium salts do not cause the oxidation of ethyl alcohol to acetaldehyde. The action of cadmium and zinc salts is ionic.—S. S. Z.

*Alcoholic fermentation. X. Fermentation is life without oxygen.* S. Kostytschew and P. Eliasberg. *Z. physiol. Chem.*, 1920, 111, 141—156.

SEVERAL species of mucus were employed for fermenting sugar under aerobic conditions, and the carbon dioxide produced through direct respiration was estimated. It was found that under the experimental conditions the organisms oxidised the sugar to an extent sufficient to cover their vital energy.—S. S. Z.

*Nitrates; Utilisation of — by moulds for the production of nitrogenous compounds.* S. Kostytschew and E. Tswetkova. *Z. physiol. Chem.*, 1920, 111, 171—200.

*Aspergillus niger* and *Mucor racemosus* reduce nitrates and nitrites to ammonia, from which they synthesise amino-compounds with the help of sugar. Nitrous acid, ammonia, and amino-compounds have been definitely observed in the intermediate stages. The nitrite-nitrogen is converted into ammonia and amino-nitrogen outside the hyphae, but is not assimilated by them as such in experiments of short duration. The reduction of nitrate to nitrite takes place, at least in *Mucor racemosus*, only in the presence of sugar.—S. S. Z.

*Sugar; Formation of — by moulds from non-sugars.* S. Kostytschew. *Z. physiol. Chem.*, 1920, 111, 236—245.

SUGAR and alcohol were formed by *Aspergillus niger* from the following substances: *d*-tartaric acid, glycerol, quinic acid, mannitol, and lactic acid. From peptone, on the other hand, no sugar was formed.—S. S. Z.

*Acetaldehyde; Formation of — in the decomposition of sugar by moulds.* C. Cohen. *Biochem. Zeits.*, 1920, 112, 139—143.

THE formation of acetaldehyde as an intermediate product was established by means of the secondary sulphite "fixation" method in the fermentation of dextrose by *Aspergillus cellulose*, *Monilia candida*, *Mucor racemosus*, *Mucor Rouzii*, and *Oidium lactis*. —S. S. Z.

*Acetaldehyde as an intermediate product in the fermentation of sugar by Lactis aerogenes.* C. Neuberg, F. F. Nord, and E. Wolff. *Biochem. Zeits.*, 1920, 112, 144—150.

ACETALDEHYDE has been found by the "fixation" method to be an intermediate product in the fermentation of sugar by *B. lactis aerogenes*.—S. S. Z.

*Sugar-cane fibre and cottonseed hulls; Acid hydrolysis of — [for production of alcohol].* E. C. Sherrard and G. W. Blanco. *J. Ind. Eng. Chem.*, 1920, 12, 1160—1162.

BAGASSE and cottonseed hulls, without previous treatment, are unsuitable for the preparation of ethyl alcohol by direct hydrolysis with acid and subsequent fermentation. Of the total sugars produced by the acid hydrolysis of these materials, only a small quantity is fermentable and the larger proportion consists of pentoses. Bagasse may prove of value as a source of xylose and furfural; in the experiments described, 12.1% of pure xylose, calculated on the quantity of dry sugar-cane fibre, was obtained.—W. P. S.

*Alcoholic strength; Tables giving — from the specific gravity. II. 25—50% of alcohol by weight.* G. Tommasi. *Ann. R. Staz. Chim.-Agrar. Sperim.*, 1917—1919, II, 3, 37—74.

TABLES are given showing percentage of alcohol by weight; percentage of alcohol by volume at 15° C.; and number of grams of alcohol in 100 c.c. at 15° C. for the specific gravities of aqueous alcoholic solutions determined at a number of different temperatures ranging from 10°/15° to 25°/15° C.

—T. H. P.

*Vinegar; Orange —. Its manufacture and composition.* H. D. Poore. *J. Ind. Eng. Chem.*, 1920, 12, 1176—1179.

BRIEF descriptions are given of two processes used to produce orange vinegar, one in a small way by the barrel or roller process and the other on a large scale by the well-known generator process. The

vinegar obtained by the latter process contains acetic acid, 3.7–4.1; fixed acid (as citric acid), 1.0–1.2; total solids, 3.82–5.26; ash, 0.52–0.79;  $P_2O_5$ , 0.035–0.053; proteins, 0.56–0.75 g. per 100 c.c. The product obtained by the barrel process is slightly higher in acidity.—W. P. S.

*Chymosin and pepsin.* Hammarsten. See XIXA.

*Yeast nucleic acid.* Steudel and Peiser. See XX.

#### PATENTS.

*Glycerol; Manufacture of — from sugar.* Verein. Chem. Werke A.-G. E.P. 133,323, 21.1.20. Conv., 22.4.16. Addn. to 133,099 (J., 1920, 608 A).

IN the process of the chief patent a sulphite is employed as alkaline substance; e.g., 1 kg. of sugar fermented at 30° C. by 100 g. of yeast in 6.6 l. of water containing 100 g. of anhydrous sodium sulphite, yields 14.3% of glycerol and 39% of alcohol; by increasing the amount of sulphite added to 120 g. and 250 g. the yield of glycerol is raised to 16% and 23.5% and that of alcohol lowered to 30% and 24% respectively. Yeast foods and catalysts such as manganese or iron sulphate may be employed, and a further improvement is effected by using in addition to sulphites a small proportion (e.g., 0.2–1% of the sugar) of salts of strongly reducing sulphur acids, such as hydrosulphites or sulphonylates. The yield of glycerol may thus under certain conditions be raised to 30% of the sugar consumed.—J. H. L.

*Yeast; Manufacture of —.* Aktieselskabet Dansk Gaerings Industri, and S. Sak. E.P. 153,667, 11.8.19.

IN the manufacture of yeast, especially by the aeration method, mash or wort of higher concentration than that in which the yeast was pitched is added in the course of propagation; the conditions of yeast growth are thus rendered more uniform throughout than when the concentration of the nutritive matters diminishes continuously. For example, the yeast may be pitched in wort (later mash drainings) of 2% Balling, and after 2–3 hrs. the main wort, of 10% Balling, may be added, continuously or intermittently, over a period of 10–11 hrs. By suitable aeration the yeast may be made to assimilate the alcohol formed, to any required extent, or the alcohol may be recovered.—J. H. L.

*Sugars; Process of producing fermentable — [from cellulosic material].* G. H. Tomlinson. U.S.P. 1,358,898, 16.11.20. Appl., 13.6.17.

THE material is heated in contact with a hydrolysing agent in a closed vessel, and after saccharification has been effected the pressure is rapidly reduced to below 1 atm. and volatile reaction products are removed by distillation.—J. H. L.

### XIXA.—FOODS.

*Meat and fish; Absorption of salt by — from a freezing mixture.* S. Schmidt-Nielsen. Tekn. Ukeblad, 1920, 67, 456. Chem.-Zeit., 1920, 45, Rep., 313.

WHEN meat and fish are immersed in sodium chloride solution at ordinary temperature they absorb considerable quantities of the salt, but when they are placed in a mixture of ice and salt at –5° to –15° C., or the liquid mixture of the same at this temperature, the absorption of salt is very small and is negligible at a depth of 2 mm. below the surface of the meat. The penetration of the salt is the greater the higher the temperature of the liquid above its freezing point.—W. P. S.

*Linseed cake and oil-free mustard residues; Composition and digestibility of —.* F. Honcamp, H. Zimmermann, and O. Nolte. Landw. Vers.-Stat., 1920, 96, 339–352.

FEEDING experiments were made on sheep with a daily ration of 600 g. of hay and 200 g. of linseed cake or 240 g. of oil-free mustard residues. The percentages of the various constituents of the two feeding stuffs digested were: organic matter, 80, 77.6%; crude protein, 83, 86.5%; nitrogen-free extract, 90, 73.6%; ether extract, 96, 88.8%; crude fibre, 35, 52.7%. These results showed that the amount of digestible protein contained in the two feeding stuffs was 27.15% in the linseed and 34.62% in the mustard. The two feeding stuffs are therefore similar in composition and digestibility.—J. H. J.

*Crude fibre [in feeding stuffs]; Determination of —.* O. Nolte. Landw. Vers.-Stat., 1920, 96, 326–337.

SEVERAL points affecting the determination of crude fibre in feeding stuffs by various methods were investigated. If the basin used was smaller than the usual 200 c.c. size, low results were obtained, and occasionally also if it was larger. Very rapid boiling also led to low results. If the strength of either the acid or the alkali was diminished below the usual 1.25%, a higher yield of crude fibre was obtained, although the percentages of carbon and hydrogen in it remained the same. The use of a stronger alkali for a shorter time caused the results to be distinctly lower. Whether fat was removed or not before determining the fibre made little difference. A large number of results are given showing the percentages of the particles of various sizes in coarsely and finely ground meals. The amount of crude fibre found in these samples tended to decrease with fine grinding.—J. H. J.

*Straw fodder; Determination of the degree of hydrolysis of —.* Von Wissell. Landw. Vers.-Stat., 1920, 96, 263–275.

THE best means of determining the digestibility of fodder prepared by digesting straw of cereals and other material rich in cellulose, with dilute alkali, is to determine the loss in weight on hydrolysis with 1% caustic soda, to apply the phloroglucinol test for lignin, and to determine the chlorine absorbed from bleaching powder.—J. H. J.

*Mustard; Determination of crude fibre in prepared —.* M. C. Albrecht. J. Ind. Eng. Chem., 1920, 12, 1175–1176.

HIGH results are obtained in the determination of crude fibre in prepared mustard unless the sample is treated previously for the removal of oil.

—W. P. S.

*Chymosin and pepsin; Action of —.* VI. Experiments on the preparation of pure stomach enzymes and observations on their action. O. Hammarsten. Z. physiol. Chem., 1919, 108, 243–286.

ON extracting the mucous membrane of the dog, pig, cow, or horse with 0.2% hydrochloric acid and mixing the clear filtered infusion with an equal volume of a saturated solution of sodium chloride, a flocculent hyaline substance is obtained. This substance contains the greater part of the enzymes of the extract. Dilutions of 1:10,000,000 were still active. It could be re-dissolved in dilute acid and reprecipitated with sodium chloride. After five precipitations the fraction still showed strong activity. If the extraction is carried out at body temperature, a precipitate is formed only some time after the addition of the sodium chloride. It is considered that the substance is denatured when extracted at 37°–38° C.—S. S. Z.

Casein. (1) Clark. (2) Clark and others. (3) Shaw. (4) Zoller. See XV.

Dextrose and starch. Quisumbing. See XVII.

Arsenic. Kohn-Abrest. See XXIII.

Nitrogen determinations. Cochrane. See XXIII.

#### PATENTS.

Grinding or pulverising food or other substances of a similar nature; Method and means for —. R. Head, and J. Baker and Sons, Ltd. E.P. 153,332, 9.6.17.

WITHIN a stationary casing is a horizontal shaft carrying a tapered cone with a space between the cone and the sides of the casing. Beaters project from the sides of the cone and engage with beaters projecting from the sides of the casing. The shaft is driven by a pulley placed externally to the casing. The material to be ground is fed by a hopper and worm into the casing at the narrow end of the cone and delivered by a feed screw to the annular space around the cone. A fan mounted on the shaft externally to the inlet end of the apparatus sends a current of air through the casing, which cools and aerates the material and aids in its discharge from the apparatus.—J. H. J.

Coffee substitutes containing caffeine; Process for the production of —. K. Lendrich. E.P. 153,971, 18.8.19. Addn. to 131,304.

IN the manufacture of coffee substitutes from raw or malted cereals by steeping in water which may contain salts, then heating by steam under pressure, and finally roasting, caffeine is added to the steeping water.—J. H. L.

Drying foodstuffs; Machine for —. N. C. Hero. U.S.P. 1,358,313, 9.11.20. Appl., 22.1.19.

A SERIES of three coaxial casings is arranged within a horizontal shell. The outermost casing tapers slightly and at its narrow end is connected with the innermost casing, the opposite end of which passes beyond the shell. The intermediate casing communicates with the interior of the shell at the broad end of the outer casing. All the casings rotate together. A feed-tube supplies the material to be dried to the innermost casing externally to the shell. A blast of air is sent through the innermost casing, and hot air is supplied to the intermediate casing.—J. H. J.

Food product [from maize] and method of preparing the same. F. G. Lorenzen, Assr. to Kellogg Toasted Corn Flake Co. U.S.P. 1,358,960, 16.11.20. Appl., 30.3.18.

STARCHY material from maize is moistened with water and converted into a flaked product which is then cooked by dry steam, dried, and granulated.—W. P. S.

Milk and cream substitutes; Process of manufacturing —. G. D. Thevenot. U.S.P. 1,359,633, 23.11.20. Appl., 24.1.19.

VEGETABLES rich in proteins, such as soya beans, are softened and sterilised by cooking, and freed from colouring matter; the solid portion, separated from the liquid, is crushed to a fine pulp, mixed with sterilised and slightly alkaline water, digested with proteolytic enzymes in presence of sodium chloride, and the resulting extract together with suspended matters is mixed with fats, oils, and sugar.—J. H. L.

Casein and vegetable albumin, including gluten; Process for the manufacture of a colloidal solution, neutral to the taste, from —. M. Monhaupt. E.P. 154,627, 20.7.17.

See U.S.P. 1,326,210 of 1919; J., 1920, 204 A.

Drying cylinders. E.P. 154,508. See I.

## XIXB.—WATER PURIFICATION; SANITATION.

Water; Sterilisation of — by chlorine gas. J. S. Arthur. J. Inst. Mech. Eng., 1920, 1127—1198.

A DETAILED account is given of the types of apparatus used for sterilising water for the Army during the war. For a filtered water less than 0.5 pt. of chlorine per million pts. of water is required for complete sterilisation, and for a neutral raw water, such as that of a large river, 3—4 pts. per million with a period of 20 mins. contact. An apparatus devised by Wallace and Tiernan (E.P. 113,105—6, 113,197—8, 113,288, and 113,374; J., 1918, 191 A, 192 A) was adopted as the standard type for the British Army. The apparatus is in two forms. The direct feed chlorinator consists of a pressure compensator, a flow measurer, and a back-pressure valve and diffuser. It delivers a steady flow of chlorine gas at a constant pressure from bottles of liquid chlorine. The accuracy is almost 100%. The solution feed chlorinator is a similar apparatus with a bubble meter in addition, in which the bubbles of gas can be seen and counted as they dissolve in the water in the meter. This water, when saturated, is injected into the bulk of water to be treated. Drawings and plans are given of the portable and stationary plants used in the Army, the process of purification adopted in each being essentially the same and consisting of five steps: Coagulation of suspended matter with aluminium sulphate and alkali; sedimentation; filtration of the supernatant water; sterilisation; de-chlorination with sulphur dioxide. A sand filter is used, and filtration takes place from the top downwards, the sand being kept covered with water; it is flushed by upward flow when it becomes clogged. The amount of chlorine to be added is determined by a preliminary test with a standard solution of bleaching powder. After the addition of chlorine the water is passed through a series of tanks for a period of 20 mins. to complete the sterilisation. For de-chlorination sulphur dioxide gas is used, delivered from a bottle of the liquid through a fine adjustment valve. The amount to be used is determined from time to time by adding potassium iodide and starch to samples of the water after treatment with sulphur dioxide; the development of a blue colour indicates excess of chlorine, and enough sulphur dioxide is added to reduce this colour to less than that produced by 0.5 pt. of chlorine per 100,000 parts of water. This small amount is left in to maintain sterility until the water is consumed, by which time it has almost disappeared and no taste is left.—J. H. J.

Water analysis [; Determination of sulphuric acid in —]. L. W. Winkler. Z. angew. Chem., 1920, 33, 311—312.

SULPHURIC acid in potable waters may be approximately estimated by observing the time at which the liquid first becomes cloudy, when to 5 c.c. of the clear sample, acidified with 2.5 c.c. of 10% hydrochloric acid, 2.5 c.c. of 10% barium chloride solution is added. The test is conveniently carried out beside a blank experiment in two test glasses standing on a black surface to facilitate observation, and the values for sulphuric acid corresponding with the time are obtained either from a table quoted in the paper, or preferably by actual tests on dilute sulphuric acid solutions of known strength from which a curve showing times and mg.  $H_2SO_4$  can be constructed. For a gravimetric estimation it is generally necessary to concentrate by evaporation unless large quantities of sulphate are present, but incorrect results are obtained unless calcium is first removed by precipitation with 0.5—1.0 g. of sodium hydroxide and 1—2 g. of sodium carbonate per litre, the precipitate being allowed to settle, and 1000 c.c. of the supernatant clear liquid

evaporated to dryness after acidification with hydrochloric acid. The residue is then dissolved in 25 c.c. of water, filtered free from silicic acid, and the sulphuric acid estimated as previously described (J., 1920, 331 A, 584 A).—G. F. M.

*Indican (indoxyl potassium sulphate); Detection of very small quantities of — in water as an aid to hygienic water analysis.* A. Jolles. Ber. Deuts. Pharm. Ges., 1920, 30, 421–442.

THE presence of indican in a water, even in the merest traces, is a certain indication of previous contamination with animal excretions, and as its detection by chemical means is very simple and not interfered with by the commonly occurring constituents of natural waters, excepting nitrites, which, however, are readily eliminated by means of Mohr's salt (ferrous ammonium sulphate), its presence or absence forms an excellent criterion for the judgment of a water from the hygienic standpoint. The test is performed as follows: 3–4 l. of the water is evaporated to 250 c.c., and if nitrites are present 3 g. of Mohr's salt is added for every 0.1 g. of nitrite per litre of the original water. The evaporation is then continued to 10 c.c., and to the filtered solution 1 c.c. of 5% alcoholic thymol or  $\alpha$ -naphthol solution, and 10 c.c. of fuming hydrochloric acid containing 5 g. of ferric chloride per litre are added. After keeping for 15 mins. with occasional shaking the indolignone colouring matter which will have been formed if indican was originally present, is extracted by careful shaking with 4 c.c. of chloroform. A reddish or bluish violet coloration of the chloroform layer, however slight, is a certain proof of the presence of indican and of the previous contamination of the water with animal excreta.—G. F. M.

*Air containing toxic gases; Purification of —.* A. Desgrez, H. Guilleminard, and A. Savès. Comptes rend., 1920, 171, 1177–1179.

VARIOUS spray fluids sprayed in with a Vermorel sprayer are recommended for neutralising different toxic gases. For chlorine a liquid containing 220 g. of sodium thiosulphate, 175 g. of sodium carbonate, and 1000 c.c. of water is recommended. For a mixture of carbonyl chloride and chlorine a 12% solution of sodium carbonate is effective. A fluid containing 240 g. of sodium polysulphide and 140 c.c. of soap-boiler's lye in 1 l. is effective in neutralising chlorine, carbonyl chloride, chloromethyl chloroformates, acrolein, bromoacetone, cyanogen chloride, chloropicrin, and benzyl chloride, bromide, or iodide.—W. G.

*Oligodynamic action of metals.* D. Acél. Biochem. Zeits., 1920, 112, 23–26.

WATER treated with metallic silver has been shown by means of ammonium sulphide and by means of potassium chromate to contain silver. It was also demonstrated that if the dissolved silver was removed the water lost its oligodynamic action (cf. J., 1920, 702 A). The author therefore concludes that the oligodynamic action of silver is due entirely to the solubility of the metal in the water.—S. S. Z.

#### PATENTS.

*Sewage sludge or the like; Method and means for reducing the percentage of water in —.* L. Linden. E.P. 153,335, 11.8.19.

SEWAGE sludge is run into settling chambers provided with upright revolving arms or with compressed air inlets, or with both, with the object of violently agitating the sludge so as thoroughly to incorporate any remains of a precipitant added previously. Additional precipitants may be added to the sludge, and after continuing the agitation for, say, five minutes, it is suddenly stopped. The

heavy sludge sinks rapidly and completely. The supernatant liquid is drawn off at the top of the chambers, and the sludge from openings at the bottom.—J. H. J.

*Waste liquors; Method of and apparatus for treating —.* C. L. Peck, Assr. to The Dorr Co. U.S.P. 1,357,587, 2.11.20. Appl., 1.12.19.

WASTE liquid containing suspended solids is submitted to a combined process of sedimentation and flotation, the deposited and floating solids being both worked mechanically towards a common discharge region, whence they are removed together.—J. H. J.

*Waste organic substances; Apparatus and process for treating —.* A. MacLachlan. U.S.P. (A) 1,359,085 and (s) 1,359,086, 16.11.20. Appl., 23.11.18 and 6.6.19.

(A) A TANK in which the material is treated is connected with a chamber in which sulphur is burned, and this chamber is connected with another which is open to the air; a steam injector is inserted in the pipe connecting the sulphur chamber with the treatment chamber, so that sulphur dioxide and air may be introduced into the material. (s) Material containing fat is subjected to the action of sulphur dioxide and steam in order to cause separation of the fat.—W. P. S.

*Insecticide.* E.P. 154,057. See XVI.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Opiums; Content of ammonium salts in various —.* J. L. Thomson. Pharm. J., 1920, 105, 550.

AMMONIUM salts in small amounts are a normal constituent of opium. The analysis of a large number of samples of Turkish opium showed an average content of 0.22% of ammonia, of Indian opium 0.21%, and of Persian opium 0.18%. The fluctuations in amount were greatest in Turkish opium, viz., from 0.09% to 0.47%.—G. F. M.

*Opium; Effects of prolonged heating and of aqueous extraction on —.* A. M. Macmillan and A. Tingle. Amer. J. Pharm., 1920, 92, 810–815.

OPIMUM when heated lost weight steadily, no sign of constancy being observed even after heating for 576 hrs. at 93°–100° C. At the same time there was a diminution in the morphine content, Persian opium losing more than 30% of its morphine in 96 hrs., but no more when the heating was continued for 288 hrs., whilst Indian opium lost only 3% in 96 hrs., but continued to lose progressively even after 576 hrs., when a 30% diminution in morphine content had been observed. The above results are at variance with those recorded by Annett and Singh (J., 1913, 315 r). The prolonged heating of crystallised morphine even for 8 days had no appreciable effect on the basicity beyond that due to the expulsion of the water of crystallisation. Morphine cannot be completely removed from opium by water extraction. Even under the drastic treatment of boiling for 16 hrs. in a Soxhlet extractor the residue still contained 2% of the total morphine originally present, whilst a further 9% had been destroyed by the boiling.—G. F. M.

*Opium assay.* A. J. Jones. Pharm. J., 1920, 105, 550–551.

THE author criticises Dott's method (J., 1920, 347 A) and suggests the modification that 10 g. of opium be triturated with 3 g. of lime and 88 c.c. of water. This will give a dilution of the opium of 1 pt. in 9 fluid pts., and if the filtrate is diluted with one-



ninth of its volume of alcohol-ether mixture and again filtered the resulting filtrate will be exactly 1 in 10 dilution. 50 c.c., representing 5 g. of the opium, is then taken for the assay. A somewhat similar scheme is adopted for the direct assay of the wet gum.—G. F. M.

*Digitalis; Chemistry of* — H. C. Hamilton. J. Ind. Eng. Chem., 1920, 12, 1180—1181.

THE author has isolated from digitalis extract two active principles, one being soluble in chloroform and the other insoluble, but there appears to be a tendency for the soluble substance to become less soluble on repeated applications of the solvent. Experiments on frogs showed that the chloroform-insoluble substance had the greater physiological action.—W. P. S.

*Gentian preparations; Preservation of* — M. Bridel. J. Pharm. Chim., 1920, 22, 411—418.

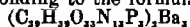
THERE was no appreciable loss of carbohydrates in powdered gentian when this was kept for nine years, but the gentiopicroin disappeared entirely; the powder, however, still contained a glucoside hydrolysed by emulsin. The sugars remained unaltered in gentian extract, but diminished considerably in tinctures during the same period; the gentiopicroin also decreased in the tinctures, a tincture prepared with 60% alcohol being quite free from this substance after the lapse of nine years. This tincture contained a glucoside having the characters of  $\beta$ -ethylglucoside.—W. P. S.

*Latex of Lactarius vellereus*. J. Zellner. Z. physiol. Chem., 1920, 111, 293—296.

THE latex of *Lactarius vellereus* consists mostly of water (80.5%) and a mixture of stearic acid and a resin (14.65%). The latter fraction can be extracted from the dried latex with ether, from which the stearic acid crystallises out on concentration and the resin remains in the mother liquor. Mannitol, dextrose, mineral substances, and proteins make up the rest of the latex.—S. S. Z.

*Yeast nucleic acid*. H. Steudel and E. Peiser. Z. physiol. Chem., 1919, 103, 42—50.

A SAMPLE of commercial yeast nucleic acid with a ratio of P:N=1:1.77 was fractionated by means of lead acetate, sodium carbonate, and barium acetate into five fractions with varying P:N ratio. As the reactions employed in the fractionation were not drastic enough to break up the nucleic acid molecule the authors conclude that the substance was a mixture of several compounds. One of these fractions with a ratio of P:N=1:2.09 on analysis gave results corresponding to the formula



for the barium salt of yeast nucleic acid.—S. S. Z.

*Nucleic acids; New methods for preparation of* — R. Feulgen. Z. physiol. Chem., 1920, 111, 257—272.

THE nucleoprotein from pancreas was prepared by digesting the minced organ with hot water, treating the aqueous extract with sodium hydroxide, filtering, and precipitating with 96% alcohol after neutralising with acetic acid. The nucleoprotein was hydrolysed with sodium hydroxide and the mixed nucleic acids precipitated with 96% alcohol; these were purified by precipitation with alcohol in alkaline solution. The two nucleic acids were separated by salting out the sodium salt of guanylic acid with sodium acetate. The tertiary salt of guanylic acid was prepared by treating the secondary sodium salt of the acid with sodium hydroxide and precipitating with 96% alcohol.—S. S. Z.

*Nucleic acids; New method of cleavage for* — H. Steudel and E. Peiser. Z. physiol. Chem., 1920, 111, 297—303.

SONIUM nucleate from the sperm of the herring was hydrolysed with calcium bisulphite solution prepared by passing sulphur dioxide through a 3% calcium chloride solution at 120°—130° C. for two hours. The resulting products of hydrolysis were precipitated with calcium acetate and purified by various precipitations. From the phosphorus and nitrogen determinations it was found that the substance thus isolated was identical with thymic acid. On hydrolysis with sulphuric acid under a reflux condenser thymine was obtained from it. The above method affords a means of preparing thymic acid with comparative ease, and the acid thus obtained shows great stability. After more than a year it retained its white colour, solubility in water, and power of reducing Fehling's solution.—S. S. Z.

*Hypnotics*. Puyal and Montagne. Bull. Soc. Chim., 1920, 27, 857—862.

WITH a view to studying the effect on their hypnotic properties of introducing a chlorine atom or a phenyl group into urethanes, the authors have prepared a series of such urethanes as follows: Ethyl- and propylchloroethyl carbinolurethanes, phenylmethyl-, phenylethyl-, phenylpropyl-, and phenylbutyl- carbinolurethanes, vinyl ethylcarbinolurethane, phenylvinyl-carbinolurethane, ethoxychloropropanolurethane, phenoxyethoxypropanolurethane, chloroethanolurethane, and cyclohexanolurethane.—W. G.

*Saccharin; Reaction of* — L. Thévenon. J. Pharm. Chim., 1920, 22, 421—422.

TEN c.c. of 0.1% sodium nitrite solution and 6 drops of dilute sulphuric acid are added to 0.1 g. of saccharin dissolved in 25 c.c. of water; after a few minutes 0.1 g. of  $\beta$ -naphthol is also added. A bright-red coloration appears at once; the colour is stable and dyes wool and silk.—W. P. S.

*Thiodiglycol; Action of* — on silver salts. C. Moureu and M. Murat. Chim. et Ind., 1920, 4, 593—596.

THIONIGLYCOL easily dissolves most silver salts, and it is not possible to precipitate the whole of the silver chloride from its solution in thiodiglycol by the addition of water. If present, therefore, thiodiglycol prevents the accurate estimation of chlorides volumetrically either by the chromate or thiocyanate methods of titration with silver nitrate. In the estimation of mustard gas (dichloroethyl sulphide) by titrating with silver nitrate the hydrochloric acid liberated by hydrolysis, the presence of thiodiglycol is therefore a source of error. The amount of the error increases with the amount of thiodiglycol present.—W. G.

*Neosalvarsan [salvarsan, etc.]; Determination of arsenic in* — A. Kircher and F. von Ruppert. Ber. Deuts. Pharm. Ges., 1920, 30, 419—421.

FROM 0.2 to 0.3 g. of salvarsan (or its derivatives) is boiled with 20 c.c. of sulphuric acid and 15 g. of potassium sulphate in a 500 c.c. round flask connected with a bulb absorption tube containing a little water. When all the organic matter has been destroyed the absorption bulb and tube are rinsed out into the flask and the liquid is diluted to about 250 c.c., boiled for 5 mins. to expel sulphur dioxide, partially neutralised with sodium hydroxide, and dilute iodine solution added, drop by drop, in presence of starch to oxidise the last traces of sulphur dioxide. The solution is then decolorised with a few drops of thiosulphate, and after saturation with powdered sodium bicarbonate, the arsenious oxide is titrated with N/10 iodine (1 c.c. = 0.003748 g. As). It is advisable to perform a blank experiment at the same time.—G. F. M.



**Triphenylarsine and diphenylarsenious salts.** W. J. Pope and E. E. Turner. Chem. Soc. Trans., 1920, 117, 1447—1452.

THE best conditions for the preparation of triphenylarsine are realised by the action of 57 g. of sodium in wire or slices on a mixture of 136 g. of chlorobenzene and 85 g. of arsenic trichloride in presence of 300 c.c. of benzene. The sodium is preferably covered first with the benzene containing 1–2% of ethyl acetate, and left for 30 mins to activate the metal, after which the other reagents are slowly run in, the mixture being finally boiled for several hours under a reflux condenser. The yield, calculated on the chlorobenzene, amounts to about 90% of the theoretical. When triphenylarsine (30.6 g.) is heated at 350° C. under atmospheric pressure with arsenic trichloride (25.5 c.c.), a mixture of phenylarsenious dichloride and diphenylarsenious chloride is produced which can be readily separated by fractional distillation *in vacuo* (cf. E.P. 142,880; J., 1920, 527 A). Diphenylarsenious chloride is readily converted into the oxide,  $[(C_6H_5)_2As]_2O$ , by boiling an alcoholic solution with alcoholic potassium hydroxide. It is a colourless crystalline solid, m.p. 89°–91° C., and on heating with hydrobromic or fuming hydriodic acid at 100° C. in a sealed tube it yields diphenylarsenious bromide, m.p. 55°–56° C., and diphenylarsenious iodide, m.p. 45°–46° C., respectively.

—G. F. M.

**Selenium monochloride; Interaction of ethylene and —.** H. W. Bausor, C. S. Gibson, and W. J. Pope. Chem. Soc. Trans., 1920, 117, 1453—1456.

ETHYLENE reacts with selenium monochloride to give  $\beta\beta$ -dichloroethyl selenide dichloride and selenium:  $2CH_2=CH_2 + 2SeCl_2 = (CH_2ClCH_2)_2SeCl_2 + 3Se$ . It seems probable that the reaction proceeds initially as with sulphur monochloride, but that the selenide is further acted on by selenium monochloride with liberation of selenium. The selenium monochloride was prepared by the action of dry hydrogen chloride on a solution of selenium in fuming sulphuric acid (15%  $SO_3$ ), and was treated with ethylene in presence of benzene as diluent. After filtering off the precipitated selenium and distilling off the benzene,  $\beta\beta$ -dichloroethyl selenide dichloride remained as white needles and after recrystallisation from benzene formed long slightly hygroscopic colourless prisms, m.p. 122.5° C. It is freely soluble in water, half the chlorine present being hydrolysed to hydrogen chloride. On passing sulphur dioxide into the cold aqueous solution an oil separates, which crystallises to a mass of colourless needles, m.p. 23°–25° C., possibly  $\beta\beta$ -dichloroethyl selenide.

—G. F. M.

**Turpentine; Thermal decomposition of — with particular reference to the production of toluene and isoprene.** S. A. Mahood. J. Ind. Eng. Chem., 1920, 12, 1152—1155.

ALTHOUGH turpentine is a possible source of toluene it does not give a sufficiently large yield either of toluene or isoprene to make it a practical source of these products under ordinary conditions.—W. P. S.

**Juniperus phænicea; Essential oil extracted from the berries of — of Sardinia.** E. Puxeddu and F. Vodret. Gaz. Chim. Ital., 1920, 50, II., 245—257.

WHEN distilled with water, the macerated berries of *Juniperus phænicea* yield 2.5% of essential oil, four-fifths of this amount distilling over during the first hour while the remainder requires about 5 hrs.; the preliminary maceration with water should last at least two days. The oil is neutral, colourless and

transparent, but turns slightly yellow under the influence of light. Its taste resembles that of camphor and it has a marked aromatic odour, while when rubbed between the hands it emits the odour of turpentine. It burns with emission of dense smoke and resinous odour. It has sp. gr. 0.8658 at 15° C.;  $[\alpha]_D^{20} = +16.84^\circ$ ; viscosity,  $\eta = 1.7259$  at 18.5° C.; solubility at 27° C., zero in 70% or 80% alcohol, 1:16 in 90% alcohol; refractive index, 1.4675 at 20° C. for ordinary light; saponification value, 14–15; acid value, 2.49; acetyl value, 35; aldehydes and phenols, absent; Maumené value, 136.8°; iodine value (Hüh), 285.53, but varying values are obtained if the amount of iodine employed in the test is varied; bromine value, 264.55. The essential oil was subjected to repeated fractional distillation under diminished pressures, the principal fraction, b. pt. 74° C. under 30 mm. pressure, having sp. gr. 0.8543 at 25° C., refractive index 1.4644, and  $[\alpha]_D^{25} = +23.94^\circ$ .—T. H. P.

**Essential oil of jasmine; Extraction of the —.** J. Nivière. Bull. Soc. Chim., 1920, 27, 862—865.

THE fact that a much greater yield of superior oil is obtained from jasmine by the method of "enfleurage" using animal fats, than by extraction with volatile solvents, has been explained by Charabot and Gatin (*La parfum chez la plante*, Paris, 1908) on the basis of the decomposition of glucosides with production of oil during the enfleurage. In support of this, it is now shown that if the jasmine flowers are submitted to a preliminary hydrolysis by acid or enzymes before extraction with solvents, the yield of oil is increased. The physical and chemical properties of the oil extracted from the hydrolysed flowers differ somewhat from those of the oil extracted from the non-hydrolysed flowers.—W. G.

**Cholesterol.** Steinkopf and others. See II A.

**Methyl alcohol.** Heuser and Schmelz. See V.

#### PATENTS.

**Pyrophosphate casein compounds; Process for the manufacture of colloidal soluble metal —.** H. R. Napp. From F. Hoffmann-La Roche & Co. E.P. 154,112, 28.2.20.

CASEIN (66 pts.) is dissolved in an aqueous solution of 112 pts. of sodium pyrophosphate, and a solution of a metal salt, e.g., 156 pts. of 4N calcium chloride solution, or 195 pts. of 23% ferric chloride solution, is added with agitation. The metal compound is precipitated by the addition of alcohol, and separated by filtration, washed, and dried at 50° C.

—L. A. C.

**Acetic acid; Manufacture of —.** British Cellulose and Chemical Manufacturing Co., M. Soller, and J. Hotz. E.P. 154,304, 21.8.19.

THE oxidation of liquid acetaldehyde to acetic acid is accomplished by using china clay, with or without the addition of sodium acetate, as a catalyst, and passing free oxygen into the agitated mixture at 10°–20° C.—D. F. T.

**Acetic acid; Apparatus for the manufacture of —.** H. W. Matheson. E.P. 154,368, 9.9.19. (cf. E.P. 132,558; J., 1919, 846 A.)

ACETALDEHYDE is treated with air in the presence of a catalyst in an aluminium-lined vessel, which contains two water-cooled metal coils for the regulation of the temperature and is fitted with two condensers, water-cooled and brine-cooled respectively, in series, the condensed product from each being returned to the reaction vessel by means of a connexion below the level of the liquid.—D. F. T.

*Acetic acid; Manufacture of.* — H. Dreyfus. E.P. 154,680, 28.8.19.

THE inorganic matter present in animal charcoal forms a satisfactory catalyst for the oxidation of liquid acetaldehyde to acetic acid; the calcium and magnesium phosphates are the most effective constituents. The residue obtained by heating animal charcoal in the air, or artificially prepared mixtures of the chief constituents of such residues, can therefore be applied to accelerate the oxidation process, preferably with the addition of sodium acetate.

—D. F. T.

*Chloroform; Manufacture of.* — M. Phillips. U.S.P. 1,359,099, 16.11.20. Appl., 13.8.20.

THE alcoholic product obtained by passing unsaturated hydrocarbons rich in propylene into sulphuric acid, and afterwards hydrolysing the alkyl sulphates, is treated with chloride of lime (bleaching powder).—J. H. L.

*Sodium and potassium formate; Production of — from calcium formate.* Elektrochem. Werke G.m.b.H., Bosshard, and D. Strauss. G.P. 325,638, 16.5.19.

ON treating calcium formate (12 mols.) with sodium or potassium sulphate (10 mols.), 96% of the latter undergoes conversion into formate; the remaining 4% of alkali sulphate is of no disadvantage in the subsequent fusion to oxalate, whilst the excess of calcium formate on interaction with alkali oxalate or carbonate yields alkali formate, the calcium simultaneously being rendered insoluble.—D. F. T.

*Perhydronaphthylphenylmethane - o - carboxylic acids; Preparation of.* — R. Willstätter. G.P. 325,714, 23.4.19.

PERHYDRONAPHTHYLPHENYLMETHANE - o - CARBOXYLIC acid,  $C_{18}H_{16}O_2$ , is obtained by the reduction of naphthoyl-o-benzoic acid with hydrogen in the presence of a catalyst, such as platinum, palladium, or nickel, or, in particular, an oxide of the last-named metal; it exists in four modifications—the  $\alpha$ -modification, rhombic tablets, m.p. 129° C.;  $\beta$ -form, rhombic tablets, m.p. 114° C.;  $\gamma$ -form, prisms, m.p. 94° C.;  $\delta$ -form, a viscous syrup. These acids are of value as substitutes for the higher fatty acids. If naphthoylbenzoic acid is heated with a zinc-copper couple and an aqueous solution of ammonia, no perhydro-acid results, the product being naphthylphenylmethane-o-carboxylic acid.—D. F. T.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

[Photographic] development; Some results of recent investigations on the theory of —. A. H. Nietz. Communication No. 100 from Eastman Kodak Research Lab. Phot. J., 1920, 60, 280—291.

THE reduction potential of a developer may be measured by the amount of bromide required to produce a given depression in the density or by the concentration of bromide against which the developer can just develop. If the straight line portions of a series of curves connecting the log exposure (log E) with the density (D) of a plate for a series of times of development with the same bromided developer be continued, they will be found to meet at a point below the log E axis. If this point be  $a$ ,  $b$ , then  $D = \gamma(\log E - a) + b$ , which also follows mathematically on the experimental proof that if density be plotted against degree of development ( $\gamma$ ) the locus is a straight line,  $D =$

$\theta(\gamma - A)$ , from which  $b = -A\theta$  and  $a = \log E - \theta$ . The values of  $a$  and  $\theta$  are found to be constant for a long range of bromide concentrations, while  $b$  is proportional to log bromide concentration over a considerable range. Plotting  $b$  against log C (concentration), if  $C_0$  is the intercept on the log C axis for the bromide concentration which just depresses the density, then  $d$  (density depression)  $= b = m(\log C - \log C_0)$ . The value of  $m$  is 0.5, practically independent of developer and of emulsion, so that  $C_0$  is a measure of the reduction potential. The values of reduction potential are given for 18 developers taking that for quinol as 1. Rate of development is found experimentally to be proportional not only to the amount of work still waiting to be done ( $D_\infty - D$ ) but also inversely to the time from first appearance of the image, which leads to a more accurate method of arriving at maximum density by plotting  $\log D_\infty / (D_\infty - D)$  against log  $t$ , choosing  $D_\infty$  by trial and error to obtain a straight line (see also Bloch, Phot. J., 1917, 57, 60; J., 1917, 553). It is suggested that, except for quinol and substituted quinols, the maximum density is higher the greater the reduction potential. The effect of bromide on maximum density is the same as the effect on density in the earlier stages of development. Maximum contrast obtainable is not affected by bromide, but the practical maximum contrast is affected by the reduction of fog. The effect of bromide on velocity of development is only in the early stages of development—the period of induction—after which the velocity is the same with or without bromide. The equations deduced for density and rate of development are utilised in the determination of speed and of maximum contrast. Speed of emulsion and fogging power of the developer are not apparently functions of the reduction potential, and fog formation does not appear to follow the same law as normal density development. Comparing the constitutional formulae of the developers studied it appears that aminophenols are the most energetic hydroxyphenols the next and amines the least; substitution of a single methyl group in the amino group or in the nucleus increases the energy, but two methyl groups are not always still more effective. The introduction of a second amino group increases the potential while conversion to a glycine or introduction of  $CH_2OH$  lowers it; these results are of partial application only as the effect of group position has not been worked out.—B. V. S.

*Phenols; Tautomerism of.* — I. Quinol. W. Fuchs and B. Elsner. Ber., 1919, 52, 2281—2286.

QUINOL is capable of reacting in its tautomeric form (as an unsaturated cyclic ketone) with sodium hydrogen sulphite. The sodium salt of cyclohexane-1,4-diol-1,2,4-trisulphonate has been isolated from the reaction product and found to function as a photographic developer, which may possibly exist in the ordinary quinol developing solution.

*Feeble conductors of electricity; A new property of substances which are —.* G. Rehoul. Comptes rend., 1920, 171, 1052—1055.

IF in a dark room a photographic plate is covered with a sheet of paper which is in turn put in contact for 24—48 hrs. with two electrodes having a potential difference of 1000 volts, on developing the plate the fibres of the paper are reproduced and the equipotential lines are roughly marked. Coarse-grained paper gives a better result than fine-grained paper. The impression on the plate is much stronger if the conductivity of the paper is increased by damping it and then allowing it to dry in the air. The effect disappears almost completely and is confined to the neighbourhood of the electrodes if the paper is perfectly dry. It is apparently necessary that the material between the

photographic plates and the electrodes shall be a feeble conductor and that it shall be heterogeneous or present superficial discontinuities such as might be caused by superficial incisions in the paper.

—W. G.

*Cyanine dyes.* Mills and Hamer. See IV.

## XXII.—EXPLOSIVES; MATCHES.

*Explosives; Velocity of decomposition of high — in a vacuum.* I. R. O. Farmer. Chem. Soc. Trans., 1920, 117, 1432—1445.

THE relative stability of high explosives can be measured by determinations of the velocity of decomposition, as indicated by the rate of evolution of gas, on heating in a vacuum. The apparatus employed consists of a lagged copper bath provided with a reflux condenser, and six orifices in the lid into which steel tubes are loosely fitted to guard against damage by explosion. They are packed round with copper filings to give good contact with the bath. Special precautions are taken to guard against failure of the condenser water supply during the prolonged heating. The glass tubes containing weighed quantities of the explosive under examination are each provided with a ground-in stopper leading to a capillary manometer tube which terminates in a cup containing sufficient mercury to fill the upright limb of the capillary. The tube is exhausted through the cup and the mercury rising in the capillary forms automatically a seal, and its height records the pressure within the tube. The apparatus is then placed in the heating bath, the material is thoroughly dried by heating to a temperature at which no measurable decomposition occurs (say, 80° C.), and again exhausted, and finally heated at the desired temperature, whilst readings of the pressure are taken from time to time from which the evolved gas volume is deduced. It was found that all explosives undergo a gradual decomposition at temperatures considerably below their ignition points. The velocity is highly affected by temperature and by the catalytic action of impurities. The trinitro aromatic compounds are in general much more stable than the nitric ester explosives. The dinitro-compounds show scarcely any measurable decomposition, whilst on the other hand the nitro-amines such as "tetryl" are less stable. The relative stability is roughly indicated by the temperatures at which the gas evolution amounts to 1 c.c. per g. in 100 hrs., viz., trinitrobenzene 190°—195° C., trinitrophenol 150°—155° C., 2,4,6-trinitrotoluene 135°—140° C., tetryl 115°—120° C., nitrocellulose 100° C. The stability of T.N.T. is not appreciably affected by the addition of small quantities of isomeric trinitrotoluenes, whilst mixtures of picric acid and T.N.T. show an even slower rate of decomposition than T.N.T. alone. The addition of an unsaturated substance (castor oil) strongly depressed the stability of both picric acid and T.N.T.—G. F. M.

*Umbrite; A new explosive, —: its use in agriculture.* G. Tommasi. Ann. R. Staz. Chim.-Agrar. Sperim., 1917—1919, II., 9, 75—94.

UMBRITE A, containing 49 pts. of nitroguanidine, 38 of ammonium nitrate, and 13 of silicon, is suitable for artillery shells, while umbrite B, containing 37.5 pts. of nitroguanidine, 49.4 of ammonium nitrate, and 13.1 of silicon, which has a greater disruptive power, serves well for bombs, for mining purposes, and for loosening the soil. These explosives are but slightly hygroscopic and retain unaltered their explosive power even when they contain a certain proportion of water.—T. H. P.

## XXIII.—ANALYSIS.

*Adsorption in analytical chemistry; Importance of —.* I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 1510—1529.

THE adsorbing action of filter paper, at least so far as electrolytes are concerned, is to be ascribed to the ash content, which functions as a calcium-permutite, in which the calcium ion may be replaced by another positive ion, which thus becomes fixed by the paper. Thus anions are not adsorbed, whilst equivalent quantities of positive ions are taken up, corresponding to the alkalinity of the ash. The capillary action of filter paper, as manifested by the sucking up of liquids and solutions, appears not to be a pure adsorption phenomenon. (Cf. J.C.S., Jan.)—S. I. L.

*Acids; Adsorption of — by filter-paper.* I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 1571—1577.

THE amount of acid taken up by filter-paper was found to be constant for each kind of paper, and to be exactly equivalent to the alkalinity of the ash. In one case the adsorption law was found to hold for several acids within certain limits of dilution. (Cf. J.C.S., Feb.)—S. I. L.

*Conductometric titrations; Application of — in analysis.* I. M. Kolthoff. Chem. Weekblad, 1920, 17, 694—701. (See J., 1920, 529A, 530A, 558A, 717A, 733A, 735A, 767A.)

LIMITS are given for the dissociation constants of acids and bases which can be determined by the conductivity method, and modifications are suggested by means of which titrations of weak and strong acids and bases, alone, or in presence of one another and of salts, etc., can be carried out. (Cf. J.C.S., Feb.)—S. I. L.

*Permanganate titrations; Catalysis of —.* P. H. Segnitz. J. Ind. Eng. Chem., 1920, 12, 1196—1197.

By the addition of manganous sulphate solution in permanganate titrations, the reaction velocity is increased, and the time required can be reduced from 35 mins. to 8 secs. in the case of hydrogen peroxide-permanganate titrations and from over 1 hr. to 6 mins. in that of cold oxalate-permanganate titrations. The accuracy of the end-point is not affected.

*Potassium; Determination of — by the Lindo-Gladding method.* H. C. Moore and R. D. Caldwell. J. Ind. Eng. Chem., 1920, 12, 1188—1189.

WHEN dealing with pure potassium salts, the same results are obtained whether the potassium platinum-chloride precipitate is washed with 80% alcohol or 95% alcohol; sodium salts, however, if present, are more soluble in the 80% alcohol, and this alcoholic solution of sodium salts has a distinct solvent action on the potassium platinumchloride, causing low results for potassium to be obtained.—W. P. S.

*Barium; Gravimetric determination of — as barium sulphate.* L. W. Winkler. Z. angew. Chem., 1920, 33, 299.

THE neutral solution of the barium salt is treated with 1 g. of ammonium chloride and 1 c.c. of N/1 hydrochloric acid, boiled, and a slight excess of ammonium sulphate solution is added. The precipitated barium sulphate may be weighed either after drying or after incineration. The weight of the dry precipitate should be decreased by 0.16 mg. and that of the ignited precipitate increased by 0.6 mg. for quantities of precipitate of about 0.3 g. —W. P. S.

**Calcium; Determination of — in the presence of phosphates.** J. F. Breazeale. J. Assoc. Off. Agric. Chem., 1920, 4, 124—134.

STARTING with, say, a plant ash containing calcium, magnesium, an excess of phosphates, and some iron, but no manganese, the ash is dissolved in dilute hydrochloric acid, the solution boiled and made slightly alkaline with ammonia. A saturated solution of oxalic acid is then added until the liquid is just acid. This acid will dissolve the phosphates of iron and magnesium and convert the calcium phosphate into calcium oxalate. The solution is again made slightly alkaline with ammonia and then slightly acid with oxalic acid. The liquid is allowed to stand in a hot place until the precipitate settles and the precipitate is dealt with in the usual manner. If manganese is present it will be precipitated as oxalate along with the calcium oxalate. In this case the precipitate, after being filtered off and washed, is dissolved in excess of hydrochloric acid, the solution is made alkaline with ammonia and a little ammonium oxalate is added. The calcium is precipitated as oxalate, while the manganese remains in solution. Calcium oxalate is not soluble in oxalic acid solution at the ordinary temperature, but is appreciably soluble in boiling 30% acid. It is not soluble in solutions of ammonium or sodium salts in the presence of oxalic acid, but it is markedly soluble in solutions of sodium nitrate, chloride, or sulphate alone at the ordinary temperature and still more so in boiling solutions.—W. G.

**Manganese in presence of phosphates; Detection of —.** T. Sabalitschka and W. Erdmann. Ber. Deuts. Pharm. Ges., 1920, 30, 443—445. (Cf. Schmidt, J., 1920, 530 A.)

A CRITICISM of Schmidt's analytical method (*loc. cit.*). In presence of an excess of barium phosphate manganese is completely precipitated as phosphate by excess of ammonia, and in the further examination of the precipitate after removal of the phosphoric acid a flocculent precipitate of manganese oxalate is obtained with ammonium oxalate which is very liable to be mistaken for calcium. In presence of calcium phosphate some of the manganese is precipitated in a similar way by ammonia, whilst the rest remains in solution with the zinc in the normal way. It is further pointed out that only by leading hydrogen sulphide into the ammoniacal solution, and not by the simple addition of ammonium sulphide, is manganese precipitated entirely as sulphide from its solutions in presence of phosphates or oxalates.—G. F. M.

**Bismuth; Gravimetric determination of — as phosphate, and its application to ore analysis.** W. R. Schoeller and E. F. Waterhouse. Analyst, 1920, 45, 435—439.

THE cold bismuth solution, containing nitric acid but no chlorides, is treated with ammonia until a slight permanent precipitate is obtained; 2 c.c. of concentrated nitric acid is then added, the solution boiled, and 10% diammonium phosphate solution is added slowly. About 40 c.c. of the phosphate solution is required for 0.2 g. of bismuth, 60 c.c. for 0.4—0.5 g. The mixture is diluted to 400 c.c. with boiling water, the precipitate collected after 15 mins., washed with hot 3% ammonium nitrate solution containing a few drops of nitric acid per litre, dried, and ignited gently. The weight of  $\text{BiPO}_4 \times 0.6865$  gives the weight of bismuth. The method may be applied to the analysis of ores; the bismuth is separated from lead by precipitation with iron wire (*cf. J.*, 1900, 389), copper, arsenic, and antimony are separated by extraction of the sulphides with sodium cyanide and sulphide, and the bismuth sulphide is converted into phosphate. —W. P. S.

**Arsenic; General method for the detection and estimation of —.** Kohn-Abrest. Comptes rend., 1920, 171, 1179—1182.

FOR the estimation of arsenic in animal or human organs, physiological fluids, or foodstuffs, the material is calcined with a mixture of magnesium nitrate and oxide, using 35 c.c. of a 20% solution of magnesium nitrate and 1 g. of the oxide, the mixture being dried at 110° C. before calcining. The arsenic in the ash is determined either by Marsh's method or by reduction to arsenite and titration with standard iodine solution.—W. G.

See also pages (A) 36, *Paraffin wax* (Bube); *Viscosity at high pressure; Compressibility of lubricating oils* (Hyde); *Viscosity* (Parsons, also Boys); *Cup and ball viscosimeter*. 39, *Acetic acid* (Grottsch). 40, *Drying agents* (Sidgwick); *Naphthalenedisulphonic acids* (Ambler). 43, *Nitrates and nitrites* (Arnd); *Potash in wood ashes etc.* (Haskins); *Potassium platinichloride* (Vürtheim); *Leucites* (Tommasi). 48, *Copper in slags* (Lathé); *Roasted blende* (De Keghel); *Lead in ores* (Simmons and others). 51, *Soya bean oil* (Newhall); *Paints* (Bailey and Baldaiefen). 52, *Antimony in rubber goods* (Collier and others); *Tannin analysis* (Wilson and Kern). 53, *Sulphuric acid in leather* (Moeller); *Chlorides in leather* (Thomas and Frieden); *Casein* (Shaw, also Zoller). 54, *Moisture in soils* (Noyes and Trost); *Soil phosphorus* (Noyes); *Moisture in fertilisers* (Lipscomb and Hutchins, also Clarke); *Available phosphoric acid* (Haskins); *Ammoniacal nitrogen* (Froidevaux and Vandenberghe). 55, *Dextrose and starch* (Quisumbing); *Maltase in yeast* (Willstätter and Steibelt). 56, *Alcoholometric tables* (Tommasi). 57, *Crude fibre* (Nolte, also Albrecht); *Straw fodder* (Von Wissell). 58, *Water analysis* (Winkler). 59, *Indican in water* (Jolles); *Opium assay* (Jones). 60, *Thiodiglycol and silver salts* (Moureu and Murat); *Saccharin* (Thévenon); *Neosalvarsan etc.* (Kircher and Von Ruppert).

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

- Barytes Consolidated, Ltd., and Elliott. Separating fines from ground materials. 749. Jan. 7.  
Bramwell. Filtration of liquids. 662. Jan. 6.  
Broadbent, and Broadbent and Sons. Centrifugal machines. 36,454. Dec. 30.  
Chenard. Fractional distillation. 167. Jan. 3. (Fr., 3.1.20.)  
Cook. Chemical and physical synthesis. 36,612. Dec. 31.  
Farbenfabr. vorm. F. Bayer u. Co. Separating or isolating organic gases etc. 464. Jan. 5. (Ger., 3.11.16.)  
Fauth. Filter plates. 801. Jan. 7. (Ger., 3.9.19.)  
Freyn, Brassert, and Co. Method of gas-washing. 365. Jan. 5. (U.S., 28.6.15.)  
Giesecke. 36. See X.  
Kampf. Washing and treating artificial materials. 390. Jan. 5. (Ger., 19.4.19.)  
Langer. 454. See II.

Lilienfeld. Manufacture of colloiddally-soluble substances and suspensions or emulsions. 736. Jan. 7. (Austria, 1.8.19.)  
 Logan. Regulating specific gravity of solutions. 731. Jan. 7. (U.S., 12.7.16.)  
 McCubbin. Treating smoke, fumes, etc. 36,900. Dec. 29.  
 Martin. Separating solids from gases. 291. Jan. 4. (Ger., 21.11.19.)  
 Martin. Conveying, and wetting material to be conveyed. 292. Jan. 4. (Ger., 24.12.19.)  
 Moulin. Apparatus for diffusion of essence or volatile liquids. 36,644. Dec. 31. (Fr., 14.6.19.)  
 Nitrogen Products Co. Furnaces. 351. Jan. 5. (U.S., 27.1.19.)  
 Norsk Hydro-Elektrisk Kvaestofaktieselskab. Apparatus for effecting continuous crystallisation. 883. Jan. 7. (Norway, 17.9.14.)  
 Ondra. Concentrating, classifying, or separating pulverulent material. 300. Jan. 4.  
 Plauson and Vielle. 36,552. See II.  
 Plauson and Vielle. Manufacture of dispersoids, colloid powder, and masses therefrom. 36,554. Dec. 31. (Ger., 29.8.18.)  
 Still. Saturators for producing solid salts by treating gases with liquid. 1097. Jan. 8. (Ger., 8.8.19.)  
 Sturtevant Co. Drying-apparatus. 353. Jan. 5. (U.S., 8.12.13.)

## COMPLETE SPECIFICATIONS ACCEPTED.

15,601 (1919). Sauer. Purifying and separating or filtering liquids. (155,609.) Dec. 31.  
 15,806 (1919). Sauer. Filtering, decolorising, and purifying liquids, juices, etc. (155,611.) Dec. 31.  
 17,931 (1919). Elliott. Removal of gases from liquids. (155,804.) Jan. 12.  
 24,704 (1919). Akkerman. Process for drying liquids. (155,927.) Jan. 12.  
 4525 (1920). Littleton. See XVIII.  
 18,030 (1920). Krupp A.-G. Acid-resisting boilers, pipes, etc. (145,732.) Jan. 12.

## II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

## APPLICATIONS.

Adam. Purification of coal gas. 296. Jan. 4.  
 Anderson and Meikle. Manufacture of briquettes. 36,336. Dec. 29.  
 Bamber and Parker. Producer-gas generators. 576. Jan. 6.  
 Bates. Flotation process of fuel production. 36,298. Dec. 29. (U.S., 23.3.20.)  
 Boocock and others. 515. See VII.  
 Chem. Fabr. Worms. 239. See III.  
 Cox. Gas plant. 36,396. Dec. 29.  
 Cravau. Coke ovens. 311. Jan. 4.  
 Daynes. Detection and measurement of gases. 1117. Jan. 8.  
 Dolbear. Treating oil shales. 218. Jan. 4.  
 Dutt and Dutt. Decolorising and treating mineral oils. 277. Jan. 4.  
 Erdmann. Obtaining paraffin and highly viscous lubricating oils from bituminous masses. 526. Jan. 6. (Ger., 22.1.18.)  
 Erdmann. Obtaining paraffin from lignite tar, coal tar, or shale tar. 727. Jan. 7. (Ger., 5.8.18.)  
 Erdmann. Treatment of bituminous tars, mineral oils, tar distillates, etc. 728. Jan. 7. (Ger., 2.9.19.)  
 Erdmann. Obtaining highly viscous lubricating oils from peat tar. 729. Jan. 7. (Ger., 9.9.19.)  
 Everett and Salerni. Apparatus for distilling carbonaceous materials. 234. Jan. 4.  
 Farbw. vorm. Meister, Lucius, u. Brünning. Fuel for internal-combustion engines. 1096. Jan. 8. (Ger., 24.3.15.)

Freyn, Brassert, and Co. 365. See I.  
 Gewerkschaft ver. Constantin der Grosse. Continuous distilling oven for making gas and coke. 1093. Jan. 8. (Ger., 7.5.19.)  
 Halbergerhütte Ges. Treating plants for filtering combustible gases. 819. Jan. 7. (Ger., 29.4.14.)  
 Langer. Lubricating-oil emulsion. 409. Jan. 5. (Austria, 9.8.16.)  
 Langer. Manufacture of an oil emulsion. 453. Jan. 5. (Austria, 16.1.17.)  
 Langer. Lubricant for cylinders of steam engines etc. 454. Jan. 5. (Ger., 3.6.20.)  
 Georges-Marion-Bergwerks- u. Hütten A.-G. Gas-producers. 36,659. Dec. 31. (Ger., 14.5.15.)  
 Plauson and Vielle. Extraction of carbonaceous substances or hydrocarbons. 36,464. Dec. 30. (Ger., 23.4.18.)  
 Plauson and Vielle. Manufacture of lubricating oils. 36,552. Dec. 31. (Ger., 12.12.19.)  
 Plauson and Vielle. Extraction of montan wax from bituminous coal. 36,464. Dec. 30. (Ger., 23.4.18.)  
 Plauson and Vielle. Obtaining and oxidising paraffins etc., and obtaining soaps. 36,553. Dec. 31. (Ger., 4.6.19.)  
 Roberts. Carbonisation of coal. 72. Jan. 3.  
 Summers. Coke ovens. 473. Jan. 5.  
 Turnbull. Briquetting coke. 1132. Jan. 8. (Ger., 28.6.16.)

## COMPLETE SPECIFICATIONS ACCEPTED.

10,713 (1919). Coune. Gas washer and purifier. (155,859.) Jan. 12.  
 13,593 (1919). Soc. Franco-Belge de Fours à Coke. Horizontal coking-ovens. (127,590.) Jan. 12.  
 20,891 (1919). Broadbridge, Edser, Stenning, and Minerals Separation, Ltd. Production of coal briquettes. (155,875.) Jan. 12.  
 23,178 (1919). Macdonald, and Densified Peat Fuel and Products, Ltd. Treatment of peat. (155,895.) Jan. 12.  
 25,092 (1919). Wallwin. Gas-producers. (155,679.) Dec. 31.  
 8327 (1920). Pfeifer. Retort furnace for use in making gas. (156,029.) Jan. 12.  
 10,186 (1920). Marks (Soc. Franco-Belge de Fours à Coke). Coke ovens. (156,034.) Jan. 12.

## III.—TAR AND TAR PRODUCTS.

## APPLICATIONS.

Anderson and Atack. Fusion of organic substances with alkalis. 36,581. Dec. 31.  
 Chem. Fabr. Worms. Manufacture of anthraquinone and its derivatives. 157, 455, 457. Jan. 3 and 5. (Ger., 18.5.18, 1 and 27.12.19.)  
 Chem. Fabr. Worms. Manufacture of oxidation products of organic compounds. 286. Jan. 4. (Ger., 15.7.19.)  
 Chem. Fabr. Worms. Manufacture of constituents of low boiling-point from tar products, resins, mineral oils, etc. 289 and 456. Jan. 4 and 5. (Ger., 23.1 and 1.3.17.)  
 Erdmann. 727—729. See II.  
 Wohl. Oxidation of hydrocarbons. 257. Jan. 4. (Ger., 22.6.16.)  
 Wohl. Catalytic oxidation of organic compounds. 258. Jan. 4. (Ger., 18.12.16.)

## IV.—COLOURING MATTERS AND DYES.

## APPLICATIONS.

British Dyestuffs Corp., Peacock, and Segaller. Manufacture of oxy and sulpho-oxy derivatives of anthraquinone. 36,536. Dec. 30.  
 Carpsel (Bayer u. Co.). Manufacture of azo dyes. 146. Jan. 3.

Chem. Fahr. Worms. 157, 455, 457. *See* III.  
 Glover and Martin. Manufacture of dyes. 575.  
 Jan. 6.  
 National Aniline and Chemical Co. Disazo dye.  
 651. Jan. 6. (U.S., 1.7.19.)  
 Plauson and Vielle. Manufacture of oil-soluble  
 dyestuffs. 688. Jan. 7.

COMPLETE SPECIFICATION ACCEPTED.

5267 (1920). Hart and Stewart. Production of  
 dyestuffs. (155,726.) Dec. 31.

V.—FIBRES; TEXTILES; CELLULOSE;  
 PAPER.

APPLICATIONS.

British Cellulose and Chem. Manuf. Co., Dickie,  
 and Palmer. Manufacture of artificial filaments,  
 threads, and films. 275—6. Jan. 4.  
 Ehrenthal. Manufacture of cotton substitutes.  
 753—4. Jan. 7. (Ger., 8.8 and 24.11.19.)  
 Fabr. de Soie Artif. de Tubize. Spinning nitro-  
 cellulose solution. 1094. Jan. 8. (Ger., 28.12.17.)  
 Harnist. Treating crude cellulose. 852. Jan. 7.  
 (Fr., 6.7.14.)  
 Heigs and Müller. Treatment of highly-ligneous  
 plants etc. 399. Jan. 5. (Ger., 13.2.14.)  
 Kampf. 390. *See* I.  
 Leysieffer. Production of plastic bodies of cellu-  
 lose ethers. 818. Jan. 7. (Ger., 10.1.20.)  
 Sinclair. Manufacture of artificial silk by the  
 viscose process. 973. Jan. 8. (Belg., 20.12.19.)  
 Stein. Utilising concentrated waste sulphite  
 liquor. 470. Jan. 5. (Ger., 18.9.19.)  
 Sturtevant Co. Paper-making. 354. Jan. 5.  
 (U.S., 11.12.13.)

COMPLETE SPECIFICATION ACCEPTED.

31,191 (1919). Gillet et Fils. Treatment of vege-  
 table fibres. (150,665.) Jan. 12.

VI.—BLEACHING; DYEING; PRINTING;  
 FINISHING.

APPLICATIONS.

Davies, Thomas, and Scottish Dyes, Ltd. Dyeing  
 piece goods. 36,374. Dec. 29.  
 Mehler Segeltuchweberei A.-G. Waterproofing  
 fabrics. 851. Jan. 7. (Ger., 23.4.16.)  
 Winkler. Waterproofing-composition. 592. Jan. 6.  
 (Switz., 24.10.18.)

COMPLETE SPECIFICATION ACCEPTED.

6541 (1920). Kereszty and Wolf. Production of  
 bleaching and disinfecting agents. (147,535.)  
 Dec. 31.

VII.—ACIDS; ALKALIS; SALTS; NON-  
 METALLIC ELEMENTS.

APPLICATIONS.

Aluminium Co. Manufacture of aluminium  
 chloride. 36,516. Dec. 30. (U.S., 26.5.20.)  
 Aschenasi. Manufacture of perborates and di-  
 sodium perphosphates. 757. Jan. 7. (Ger., 27.11.18.)  
 Aschenasi. Manufacture of perborates. 758.  
 Jan. 7. (Ger., 27.11.18.)  
 Blanc and Jourdan. Extraction of potash from  
 leucite or leucitic rocks. 471. Jan. 5. (Ital., 5.1.20.)  
 Boocock, Wyld, and Holmes and Co. Recovery of  
 ammonia from ammoniacal liquor. 515. Jan. 6.  
 Burke and others. 414. *See* XIII.  
 Chem. Fahr. Worms. Manufacture of metal salts.  
 155. Jan. 3. (Ger., 13.3.18.)  
 Harger, and Woodcraft Manuf. Co. Manufac-  
 ture of hydrogen and mixtures of hydrogen and  
 nitrogen. 36,456. Dec. 30.

\*Koppers Co. Manufacture of ammonium sul-  
 phate. 36,664. Dec. 31. (U.S., 10.5.18.)  
 Nitrogen Corp. Synthesis of ammonia. 732.  
 Jan. 7. (U.S., 7.1.20.)  
 Nitrogen Products Co. Fixation of atmospheric  
 nitrogen. 352. Jan. 5. (U.S., 5.7.19.)  
 Norsk Hydro-Elektrisk Kvaelfstofaktieselskab.  
 Removing solid nitrogen oxides from refrigerating  
 devices. 882. Jan. 7. (Norway, 14.10.18.)  
 Norsk Hydro-Elektrisk Kvaelfstofaktieselskab.  
 Manufacture of concentrated nitrous gases. 884.  
 Jan. 7. (Norway, 6.10.15.)  
 Norsk Hydro-Elektrisk Kvaelfstofaktieselskab.  
 Converting nitrous gases into concentrated nitric  
 acid. 885. Jan. 7. (Norway, 22.5.15.)  
 Shimadzu. Lead oxides, and manufacture of  
 same. 36,519. Dec. 30.  
 Soc. l'Air Liquide. Synthesis of ammonia. 36,533.  
 Dec. 30. (Fr., 30.12.19.)  
 Still. 1097. *See* I.

COMPLETE SPECIFICATIONS ACCEPTED.

11,184 and 11,186 (1919). Desachy. *See* XIII.  
 17,706 (1919). Barbet et Fils et Cie. Purifica-  
 tion or argon and other rare gases of the atmos-  
 phere. (129,989.) Jan. 12.  
 23,505 (1919). Duparc and Urfer. Direct  
 oxidation of ammonia. (133,041.) Jan. 12.  
 25,545 (1919). Mackay. Manufacture of lead sul-  
 phate. (155,945.) Jan. 12.  
 26,231 and 28,489 (1919). Hood. Purification of  
 sulphur. (155,692.) Dec. 31.  
 32,397 (1919). Norsk Hydro-Elektrisk Kvaelfstof-  
 aktieselskab. Production of concentrated nitrous  
 gases and nitric acid. (137,081.) Jan. 12.  
 4369 (1920). New Jersey Zinc Co. *See* XIII.  
 8471 (1920). Soc. l'Azote Française. Manufac-  
 ture of ozone. (140,777.) Jan. 12.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Emery. Manufacture of pottery. 36,447. Dec. 30.  
 Rebuffat. Manufacture of refractory articles.  
 446. Jan. 5. (Ital., 27.2.20.)

COMPLETE SPECIFICATION ACCEPTED.

5760 (1920). Norton Co. Abrasive stone.  
 (139,502.) Jan. 12.

IX.—BUILDING MATERIALS.

APPLICATION.

Dyson. Cements. 118. Jan. 3.

X.—METALS; METALLURGY, INCLUDING  
 ELECTRO-METALLURGY.

APPLICATIONS.

Agostini. Solder for aluminium etc. 658. Jan. 6.  
 (Fr., 5.1.20.)  
 Giesecke. Sintering processes. 36. Jan. 3.  
 (Ger., 20.12.17.)  
 Gregory, and Soc d'Utilisation des Combustibles  
 Pulvérisés. Roasting minerals. 190. Jan. 4.  
 (Fr., 19.10.20.)  
 Hanemann. Plating metal objects with steel.  
 1099. Jan. 8. (Ger., 22.12.17.)  
 Keller. Alloys, and resistance grids formed  
 thereof. 123. Jan. 3. (U.S., 3.1.20.)  
 Koppers. Operating cupola and blast furnaces.  
 624—5, 832. Jan. 6 and 7. (Ger., 27.4.18, 28.3.19,  
 23.10.18.)  
 Linnmann. Cupola furnaces. 189. Jan. 4.  
 (Ger., 22.4.18.)  
 Mathesius. Lead alloys. 485. Jan. 5. (Ger.,  
 7.1.20.)

- Mawer, Painton, and Parr. Annealing and hardening high and low carbon steels etc. 64. Jan. 3.  
Schütz. Removing carbon from iron etc. 472. Jan. 5. (Ger., 11.1.18.)

## COMPLETE SPECIFICATIONS ACCEPTED.

- 5400 (1917). British Thomson-Houston Co. General Electric Co.). Metals and their manufacture. (155,851.) Jan. 12.  
14,865 (1917). Head. Production of tungsten powder. (155,600.) Dec. 31.  
15,772 (1919). Kiscock. Manufacture of alloy steel. (131,877.) Jan. 12.  
27,784 (1919). Akt. Ferrolegeringar. Production of manganese or its alloys. (135,186.) Jan. 12.  
5532 (1920). Raworth (Dean). Soldering or coating aluminium or its alloys. (156,019.) Jan. 12.  
9633 (1920). Alexander (Metal and Thermit Corp.). Formation of tungsten alloys. (155,739.) Dec. 31.  
17,760 (1920). Goldberg. Electrolytically separating copper from nickel. (145,600.) Jan. 12.  
24,343 (1920). Krupp A.-G. Separating slags containing iron from fuel residues. (150,333.) Jan. 12.

## XI.—ELECTRO-CHEMISTRY.

## APPLICATIONS.

- Reed. Electric furnace. 36,449. Dec. 30. (U.S., 27.3.20.)  
Siemens-Schuckertwerke. Insulating-materials. 137,985—6. Jan. 5 and 8. (Ger., 20.10.17, 14.12.17, 3.1.18.)  
Urbach. Electric batteries. 36,668. Dec. 31.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 21,187 (1919). Arosio. Insulating-material. (131,907.) Jan. 12.  
25,505 (1919). Willard. Paste for storage battery plates. (155,944.) Jan. 12.  
8471 (1920). Soc. l'Azote Franç. See VII.  
17,760 (1920). Goldberg. See X.  
24,854 (1920). Haglund. Electrolytic tanks with diaphragm cells. (151,260.) Jan. 12.

## XII.—FATS; OILS; WAXES.

## APPLICATIONS.

- Byk Guldenwerke Chem. Fabr. 295. See XX.  
Earp. Soap manufacture. 36,582. Dec. 31.  
Fauth. Extraction of oil from oil seeds etc. 1022. Jan. 8. (Ger., 5.6.19.)  
Granichstaden. Production of aroma of lard in artificial edible fats. 127. Jan. 3. (Austria, 18.6.15.)  
Oelwerke Stern - Sonneborn. Recording automatic apparatus for testing oils and fats. 995. Jan. 8. (Ger., 24.2.19.)  
Pech. Soap. 142. Jan. 3. (Fr., 9.1.20.)  
Plauson and Vielle. Refining oils or fats. 689. Jan. 7.  
Plauson and Vielle. 36,553. See II.  
Schneider. Extracting oil from rape seed etc. 777. Jan. 7. (Ger., 25.1.19.)

## COMPLETE SPECIFICATIONS ACCEPTED.

- 17,880 (1919). Scott and Co., and Macgregor. Extraction of fat, wax, oil, gelatin, etc. (155,863.) Jan. 12.  
18,538 (1919). Jackson (Nordiske Fabr. De-No-Fa). Producing soap powders with a high percentage of liquid fats and oils. (155,866.) Jan. 12.  
791 (1920). Green. Manufacture of margarine. (156,000.) Jan. 12.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

## APPLICATIONS.

- Akt.-Ges. f. Anilinfabr. Production of oil-colours, varnishes, etc. 283. Jan. 4. (Ger., 18.12.15.)  
Burke, Jones, and Matthews. Manufacture of lithopone, zinc sulphide, and ammonia. 414. Jan. 5.  
Chem. Fabr. Worms. Manufacture of printing and stamping colours. 154. Jan. 3. (Ger., 31.12.17.)  
Chem. Fabr. Worms. 289 and 456. See III.  
Chem. Fabr. Worms. Manufacture of artificial resins. 664. Jan. 6. (Ger., 31.3.19.)  
Chem. Fabr. Worms. Manufacture of phenol condensation products. 804. Jan. 7. (Ger., 9.9.18.)  
Harger, Richards, and Woodcraft Manuf. Co. Manufacture of paints, polishes, etc. 11. Jan. 3.  
Plauson and Vielle. Manufacture of plastic masses. 36,549. Dec. 31. (Ger., 15.3.19.)  
Plauson and Vielle. Manufacture of paintable compositions. 36,561. Dec. 31. (Ger., 23.4.18.)  
Plauson and Vielle. Manufacture of resinous condensation products. 36,563. Dec. 31. (Ger., 24.5.18.)  
Porter and White. Paint. 415. Jan. 5.  
Reeve. Rust-proof and metal preservative paint. 67. Jan. 3.  
Shimadzu. 36,519. See VII.  
Watson. Treating mineral-stained barytes to produce a pure white. 36,286. Dec. 29.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 11,184 and 11,186 (1919). Desachy. Production of anhydrous zinc sulphide. (126,625 and 126,627.) Jan. 12.  
23,379 and 23,380 (1919). Jaloustre, Kheifetz, and Warchafsky. Production of condensation products of phenols and aldehydes. (138,061 and 139,147.) Dec. 31 and Jan. 12.  
4369 (1920). New Jersey Zinc Co. Treating zinc oxide. (138,924.) Jan. 12.  
9308 (1920). Moureu and Dufraisse. Condensation products of acrolein with phenols. (141,059.) Dec. 31.  
13,930 (1920). Scholz and Tiedemann. Working up linoleum scraps into new linoleum. (143,561.) Dec. 31.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

## APPLICATIONS.

- Fitzgerald. Production of substitutes for vulcanite, horn, etc., from blood. 36,476. Dec. 30.  
Plauson and Vielle. 36,457—8. See XX.  
Plauson and Vielle. Manufacture of rubber-like substances. 36,459—36,460. Dec. 30. (Ger., 31.10 and 2.12.18.)  
Plauson and Vielle. Manufacture of rubber and ebonite substitutes. 36,555—6. Dec. 31. (Ger., 25.2.18 and 13.12.19.)  
Plauson and Vielle. Reclaiming waste rubber. 36,562. Dec. 31. (Ger., 23.4.18.)

## XV.—LEATHER; BONE; HORN; GLUE.

## APPLICATIONS.

- Chem. Fabr. Worms. Tanning skins. 61. Jan. 3. (Ger., 16.11.18.)  
Chem. Fabr. Worms. Manufacture and use of tanning materials. 665 and 813. Jan. 6 and 7. (Ger., 20.9.16 and 25.10.16.)  
Chem. Fabr. Worms. Tanning hides. 666. Jan. 6. (Ger., 23.9.16.)  
Chem. Fabr. n. Asphaltwerke A.-G. Manufacture and use of tanning materials. 288. Jan. 4. (Ger., 1.9.16.)  
Croad, Knowles, and McArthur and Co. Manufacture of tanning agents. 53—5. Jan. 3.



Niessen. Extraction of glue from raw materials. 626-8. Jan. 6 (Ger., 27.11 and 8.12.19.)  
 Railland. Manufacture of tannic extracts. 637. Jan. 6. (Fr., 27.9.17.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,780 (1919). Scott and Co., and Macgregor. *See XII.*  
 22,717 (1919). Barber and Barker. Tanning chrome leather. (155,887.) Jan. 12.

### XVI.—SOILS; FERTILISERS.

#### APPLICATION.

Plauson and Vielle. Manufacture of phosphatic manures. 36,465. Dec. 30. (Ger., 5.11.19.)

### XVIII.—FERMENTATION INDUSTRIES.

#### APPLICATION.

Plauson and Vielle. Improving colour, taste, and digestibility of raw yeast. 36,565. Dec. 31. (Ger., 16.3.19.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

9525 (1920). Littleton. Air-pressure filtering apparatus for pressing yeast etc. (155,738.) Dec. 31.  
 13,177 (1920). Fazi. Brewing or preserving beer or other fermented liquors. (143,506.) Dec. 31.

### XIX.—FOODS; WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

A.-G. vorm. Haaf u. Co. 663. *See XX.*  
 Bleicken. Apparatus for producing distilled water. 121—2. Jan. 3.  
 Granichstaden. 127. *See XII.*  
 Mardou. Antiseptic and disinfectant. 149. Jan. 3. (U.S., 20.6.18.)  
 Moore. Triple-purification water stills. 1015. Jan. 8. (U.S., 17.2.16.)  
 Schweizer. Conservation of vegetable materials. 36,671. Dec. 31. (Switz., 20.10.19.)  
 Smits. Water-purifying apparatus. 1116. Jan. 8. (Hungary, 25.3.16.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

15,601 and 15,606 (1919). Sauer. *See I.*  
 15,603 (1919). Sauer. Sterilising and purifying water. (155,610.) Dec. 31.  
 791 (1920). Green. *See XII.*  
 6541 (1920). Kereszty and Wolf. *See VI.*

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### APPLICATIONS.

A.-G. vorm. Haaf u. Co. Manufacture of healing and nutritive products. 663. Jan. 6. (Ger., 2.4.14.)  
 Anderson, and Boot's Pure Drug Co. Manufacture of derivatives of 3,3'-diamino-4,4'-dihydroxy-arsenobenzene dihydrochloride etc. 815. Jan. 7.  
 Boehringer Sohn. Production of  $\alpha$ -lobelin. 114. Jan. 3. (Ger., 21.9.16.)  
 Byk Guldenwerke Chem. Fabr. Distillation process for recovering fatty acids from fatty acid mixtures. 295. Jan. 4. (Ger., 27.6.16.)  
 Chem. Fabr. Flora. Manufacture of silver thioglycolate of sodium. 36,377. Dec. 29. (Switz., 12.11.19.)  
 Farbenfabr. vorm. F. Bayer u. Co. 464. *See I.*  
 Farb. vorm. Meister, Lucius, u. Brünig. Manufacture of a complex anrothiosalicic acid. 1100. Jan. 8. (Ger., 13.10.15.)  
 Haddock. Manufacture of lactylsalicylic acid. 36,285. Dec. 29.  
 Inray (Soc. Chem. Ind. Basle). Manufacture of therapeutic preparations from blood. 460. Jan. 5.

Plauson and Vielle. Manufacture of diolefines and polymerisation products. 36,457. Dec. 30. (Ger., 23.4.18.)

Plauson and Vielle. Manufacture of vinyl compounds and polymerisation products. 36,458. Dec. 30. (Ger., 24.5.18.)

Plauson and Vielle. Manufacture of vinyl halides. 36,461. Dec. 30. (Ger., 9.9.18.)

Plauson and Vielle. Manufacture of alkyl ethers of vinyl alcohols. 36,462. Dec. 30. (Ger., 24.5.18.)

Plauson and Vielle. Manufacture of diolefines and derivatives. 36,463. Dec. 30. (Ger., 8.12.19.)

Plauson and Vielle. Manufacture of hexamethylenetetramine. 36,548. Dec. 31. (Ger., 9.9.19.)

Plauson and Vielle. Manufacture of low-boiling chlorinated hydrocarbons. 36,551. Dec. 31. (Ger., 8.12.19.)

Plauson and Vielle. Manufacture of diols and diolefines. 36,557. Dec. 31. (Ger., 18.9.16.)

Plauson and Vielle. Oxidation of acetaldehyde to acetic acid. 36,558. Dec. 31. (Ger., 5.7.18.)

Plauson and Vielle. Manufacture of acetaldehyde or acetic acid. 36,559 and 36,564. Dec. 31. (Ger., 6.9.18, 28.1.19.)

Plauson and Vielle. Manufacture of formaldehyde and methyl alcohol. 36,560. Dec. 31. (Ger., 9.9.19.)

Rupe. Manufacture of camphylcarbinol. 1101. Jan. 8. (Ger., 2.6.16.)

Soc. Franç. des Distilleries de l'Indo-Chine. Manufacture of fatty acids of high melting-point starting from butyl alcohol. 822. Jan. 7. (Fr., 13.10.19.)

Wade (Deuts. Gold- u. Silber-Scheide-Anstalt). Manufacture of acetone, acetaldehyde, and acetic acid. 518—521. Jan. 6.

Wohl. 257—8. *See III.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

32,012 (1919). Chem. Fabr. vorm. Sandoz. Isolation of the principal alkaloid of ergot. (140,056.) Jan. 12.

13,158 (1920). Soc. Chim. Usines du Rhône. Manufacture of normal butyl *p*-aminobenzoate. (148,743.) Dec. 31.

14,465 (1920). Soc. Chim. Usines du Rhône. Preparation of dialkyl-amino-ethyl derivatives of theochromine. (155,748.) Dec. 31.

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### APPLICATIONS.

Dye Impression Photos, Ltd., and Edridge. Photographic films and plates. 36,415. Dec. 29.

Colour Photography, Ltd., Shepherd, and Thomson. Colour photography. 383. Jan. 5.

Graphische Kunstanstalten F. Bruchmann A.-G., and Kühn. Photographic printing processes. 1090. Jan. 8. (Ger., 13.11.15.)

Lnhoshey. X-ray photography. 424. Jan. 5.

#### COMPLETE SPECIFICATION ACCEPTED.

23,256 (1919). Christensen. Manufacture of coloured photographic pictures. (133,034.) Jan. 12.

### XXII.—EXPLOSIVES; MATCHES.

#### COMPLETE SPECIFICATION ACCEPTED.

22,775 (1919). Manuelli and Bernardini. Explosives. (155,627.) Dec. 31.

### XXIII.—ANALYSIS.

#### APPLICATIONS.

British Thomson-Houston Co., Millner, and Wheat. Photometers. 36,362. Dec. 29.

Daynes. 1117. *See II.*

Oelwerke Stern-Sonneborn. 995. *See XII.*

**I—GENERAL; PLANT; MACHINERY.**

**Boiler plants; Exact data on the performance of steam.**—Average figures for the performance of some different types of steam boilers. D. Brownlie. Engineering, 1920, 759—761, 797—799.

The results given were obtained both from 12-br. tests and long check tests (one week), and as the results of the short period and long period tests were in excellent agreement they may be regarded as trustworthy, in spite of their disagreement with the figures usually put forward in text-books and

glass tubes, below which is placed a reservoir. The apparatus is a Geissler pump and then sulphuric acid is drawn into the cylinder by an automatic control (Chem. Soc., 1919, 41, 53), which has a valve absorbing vapours, forcing the non-absorbable gas from the apparatus, and moistening the glass. The control then withdraws the acid into a reservoir, which improves the vacuum. The process goes on repeatedly until the evaporation or concentration is completed.—J. F. S.

*Dialysis or extraction.* Mann. See XXIII.

	Cylindrical boilers (" Lancashire," etc.).		Water-tube boilers.		Small cylindrical boilers. Average.	Small vertical boilers. Average.
	Average plants (85% of total).	Good plants (76% of total).	Average plants (85% of total).	Good plants (8% of total).		
Coal burnt per boiler per hour .. lb.	864.7	1059.8	2938.3	2859.9	846.8	112.75
Coal burnt per sq. ft. grate area per hour .. lb.	22.7	27.9	20.9	20.4	17.3	—
Water evaporation per sq. ft. grate area per hr., lb.	151.3	223.7	147.2	160.9	101.7	—
Water at 110° F. evaporated per lb. of coal ..	6.65	8.02	7.01	7.87	5.86	5.25
Water from and at 212° F. evaporated per lb. of coal ..	7.62	9.23	8.12	9.11	6.71	6.01
Temperature of water after economisers .. °F.	230	335	105	225	no econ.	no econ.
Leaving due to economisers .. °F.	11.0	20.4	7.4	10.4	nil	nil.
Draught in chimney base or fan inlet, in. water gauge ..	0.75	2.00	0.50	0.65	0.50	0.30
Temperature of gases leaving boiler .. °F.	600	650	475	450	590	800
Temperature of gases at chimney base or fan inlet .. °F.	450	310	325	300	590	800
Analysis of feed-water:—						
Permanent hardness .. deg.	9	5	6	3	9	9
Temporary hardness .. deg.	2	0	2	0	2	2
Percentage CO <sub>2</sub> in gases leaving the furnaces ..	7.5	12.0	6.0	12.5	5.0	5.0
Average gauge pressure .. lb.	75	159	155	160	70	79
Amount of superheat .. °F.	nil	170	162	279	nil	nil
Steam or power used auxiliary to the production of steam .. %	2.5	2.5	2.0	1.5	nil	nil
Efficiency:—						
Net working .. %	60.0	79.0	69.2	81.9	54.1	48.4
Boilers only .. %	54.7	59.5	60.3	65.8	54.1	48.4
Economisers only .. %	6.8	15.3	4.9	7.6	nil	nil
Superheaters only .. %	nil	6.2	5.4	9.7	nil	nil
Cost in coal to evaporate 1000 galls. of water .. d.	332.6	260.9	305.3	272.0	365.2	408.1
Coal bill for 20,000,000 galls. evaporation .. £	27,472	22,748	25,688	22,920	31,886	35,000

by manufacturers. The figures given in the table are averaged from the results of the investigation of nearly 500 steam boiler plants in many different industries, and are calculated to a basis of:—Coal of 12,000 B.Th.U. and 10.5% ash at 40s. per ton; feed water temperature before economisers 110° F. (43° C.). Size of Lancashire boilers:—30 ft. x 8 ft., with grates 6 ft. x 3 ft. 2 in. Size of tubular boilers:—Rated evaporation 20,000 lb. of water per hr., heating surface about 5250 sq. ft., with grates 4 ft. x 5 ft.—B. M. V.

**Distillation and rectification.** L. Gay. Chim. et Ind., 1920, 4, 735—748. (Cf. J., 1920, 287 A, 701 A).

The facts established in previous communications (loc. cit.) for binary mixtures are applied to mixtures containing three constituents; the complex conditions etc. prevailing in the case of a ternary mixture are indicated graphically and algebraically, and certain rules concerning the minimum and maximum of heat compatible with the satisfactory working of a rectifying column are given.

—W. P. S.

**Sulphuric acid concentrator and vacuum pump.** O. Maas. J. Amer. Chem. Soc., 1920, 42, 2571—2574.

The liquid to be concentrated is placed in bulbs connected with a large cylindrical vessel filled with

**PATENTS.**

**Producing cold; Apparatus for —.** Soc. des Condenseurs Delas. E.P. 146,094, 5.6.20. Conv., 25.6.19.

The first stage of aspiration and re-compression of the vapour of water used as a refrigerant is effected by means of a rotating fluid-friction pump from which the vapour may pass direct to a condenser, or first through a steam ejector, the combined water vapour from the latter passing to the condenser. The live steam before passing to the ejector or the combined steam leaving the ejector may be used in a steam turbine which drives the fluid-friction pump. The latter may conveniently consist of a number of discs rotating between close-fitting (not rubbing) fixed spiral guides.—B. M. V.

**Heat exchange apparatus [for gases].** H. Nielsen and F. D. Marshall. E.P. 154,308, 21.8.19.

A number of flat boxes connected by short pipes, carrying one gas, are arranged zig-zag fashion inside a casing which is provided with dampers to guide the other gas in a longer or shorter path, generally in an opposite direction to that of the first gas. The outer casing may also be provided with scrapers, operated from outside, to keep the outer surface of the boxes clean and with a bopper-like bottom from which any dust that may settle can be removed.—B. M. V.

*breaking, screening, washing, and assorting coke, pan breeze, furnace waste, and the like; Continuously acting apparatus for —.* E. J. A. Shaw and H. Blakeley. E.P. 154,376, 17.9.19.

A LONG rotating cylinder has a number of zones with perforations increasing in size progressively from the feed end; the finest material passing through the holes in the first zone drops straight to a hopper, but material passing through the second and subsequent zones drops into revolving drums concentric with and larger than the revolving cylinder, and having longitudinal troughs attached to their internal circumference and extending through their end walls. When the material collected in these troughs is raised to a suitable height it is subjected to washing by jets of water, so that the lighter material is washed over the ends of the troughs, while the heavier material remains in them and is carried round about another half revolution and is then discharged to a separate compartment.—B. M. V.

*Grinding ores, minerals, stones, and the like; Machines for —.* J. R. Broadley. E.P. 154,434, 14.11.19.

A BALL, pebble, or tube mill is provided with two additional compartments beyond the grinding compartment. The pulp passes from the grinding compartment to the first auxiliary compartment through a perforated diaphragm, and is then lifted from the first auxiliary to the second auxiliary or classification compartment by means of a scoop, so that the pulp level is higher in the classification compartment than in the first auxiliary compartment. Slime is discharged from the classification compartment through a hollow trunnion, the internal opening of which is obstructed by a circular baffle of smaller diameter than the interior of the mill, thus forming a weir to retain sand. The sand is lifted by guides and returned direct from the classification compartment to the grinding compartment through an axial passage.—B. M. V.

*Pulverising machine.* C. E. Brainard. U.S.P. 1,360,364, 30.11.20. Appl., 8.1.20.

A CYLINDRICAL casing is divided transversely into two parts, which may be brought closer together by means of a separating ring the thickness of which can be reduced to compensate for wear of the grinding surfaces. The grinding surfaces converge radially and are removable, and enclose a floating crushing ring, the sides of which converge radially to correspond with the grinding surfaces.—W. F. F.

*Crushing mill.* T. J. Sturtevant, Assr. to Sturtevant Mill Co. U.S.P. (a) 1,360,465 and (b) 1,360,468, 30.11.20. Appl., 11.7.17 and 15.6.17.

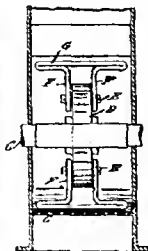
(A) A CRUSHING mill comprises a crushing ring co-operating with revolving rollers mounted on a carrier. Pressure is transmitted from one roller to another by an equinising ring. (a) The carrier for the rollers is mounted on a cover which is hinged to the casing, and spring-pressed members are mounted on the casing to transmit pressure through the carrier to the rollers. These members and the carrier automatically separate when the cover is opened and engage when it is closed.—W. F. F.

*[Grinding] mill; Combination — and process of efficient operation thereof.* P. C. Van Zandt, Assr. to Allis-Chalmers Manufacturing Co. U.S.P. 1,361,205, 7.12.20. Appl., 2.10.16.

MATERIAL is ground in the primary grinding chamber of a tube mill having two grinding chambers, and then transferred to the secondary grinding chamber. The relative volumetric capacities of the two chambers are then changed while the

normal grinding level is maintained, and the grinding operation is continued.—W. F. F.

*Reducing mill.* E. B. Campbell, Assr. to Williams Patent Crusher and Pulverizer Co. U.S.P. 1,361,679, 7.12.20. Appl., 10.2.19.



REVOLVING hammers are pivoted at E to supports, D, carried by the rotating shaft, C. Each hammer consists of shanks, F, connected to a head, G, the thickness of which between the shanks is less than the thickness of the overhanging portions. The thicker parts effect a crushing action and the thinner part a slicing action.—W. F. F.

*Ball mill.* F. E. Marcy. U.S.P. 1,362,334, 14.12.20. Appl., 10.3.19.

A ROTARY grinding mill has one of its walls in the form of a number of rolled bars, spaced apart and forming a grate. The bars are welded together at their ends at points beyond the range of the grinding bodies within the chamber.—W. F. F.

*Furnaces for heat-treating materials.* W. A. Russell, and The Manchester Furnaces, Ltd. E.P. 154,521, 29.7.20.

THE furnace is somewhat similar to that described in E.P. 3339 of 1914 (J., 1914, 1014), but the inlets for flame consist of channels cut in the underside of the roof, alternating with one another on opposite sides; and the outlets for burnt gases are situated both at the mouth and back of the furnace.—B. M. V.

*Furnaces [; Recuperative —].* A. Smallwood. E.P. 154,866, 24.6.19.

THE furnace is of similar construction to that described in E.P. 125,224 (J., 1919, 372A), but is adapted to heat materials that fill the chamber more or less completely. Combustion is arranged to take place before the gases enter the chamber, and to regulate the distribution of heat; the outlets, which are distributed along the side of the furnace, can be individually regulated.—B. M. V.

*Oven.* O. S. Sleeper, Assr. to Buffalo Foundry and Machine Co. U.S.P. 1,362,151, 14.12.20. Appl., 9.5.17.

A RETORT extends across the oven heating chamber and is provided with a number of longitudinal heating passages arranged in a vertical row and extending between inlet and outlet manifolds on opposite sides of the retort. Drying compartments, arranged in a vertical row, are disposed transversely to the heating passages and alternate with the latter. The drying compartments are permanently disconnected from the heating passages and are accessible through an opening in the side of the retort. A vertical baffle, extending above and below a heat delivery conduit from a furnace, is situated in the inlet manifold between the heat delivery conduit and the adjacent heating passages of the retort.—J. S. G. T.

*Muffle-kiln; Continuous —.* H. Webster. U.S.P. 1,362,153, 14.12.20. Appl., 27.2.20.

A CONTINUOUS muffle-kiln comprises a series of heating chambers, separated by a solid wall and communicating each with its respective furnace arranged alongside the kiln. One or more of the chambers may be provided with muffles. Hot air flues extending through the roof of the muffle into the heating chamber open downwards through the

nuffle floor and are connected with the main flue by longitudinal passages below the floor of the nuffle.—J. S. G. T.

**Heating furnace.** G. J. Hagan and J. M. Hopwood. U.S.P. 1,362,296, 14.12.20. Appl., 14.10.19.

AN annular chamber subtending an angle of less than  $360^\circ$  at its centre is provided with charging and discharging doors adjacent to its respective closed ends. Articles to be treated are placed upon an annular hearth movable in a counter direction to the flow of heat and products of combustion. Means for heating the furnace are disposed near the discharge end, and a connexion is made from the furnace to the stack near the charging end.

—J. S. G. T.

**Furnace.** G. J. Hagan. U.S.P. 1,362,297, 14.12.20. Appl., 14.5.20.

THE bridge wall of a heating furnace is provided with a passage having its inlet communicating with a point near the bottom of the heating chamber, and its outlet so arranged that the products of combustion flow across it, thereby causing a flow of gases from the heating chamber through the passage.—J. S. G. T.

**Kiln.** C. D. Vezey and C. Mariner. U.S.P. 1,362,068, 14.12.20. Appl., 13.1.19.

AN inclined shaft kiln is connected at the top with a return flue adjacent to the lower inclined wall of the kiln, the lower end of the return flue being connected with a draught flue. The connexion between the kiln and return flue is controlled by a damper, and controlled openings between the kiln and flue are provided at intervals along its length.

—W. F. F.

**Centrifugal machine for [continuously] filtering or extracting liquids, semi-liquids, and other substances.** J. McIntyre. E.P. 154,641, 30.5.19.

THE apparatus consists of a central feed cylinder around which are arranged filtering cylinders. In addition to the high-speed rotation of the whole, the filtering cylinders rotate about their own axes at a lower speed. Vertical lines of holes are provided in the inner feed cylinder opposite the nearest points of the filtering cylinders, which are covered outside with cloths or other filtering medium and are provided internally with diametral baffles to catch the liquid which passes through into the inside of the cylinders and deflect it downwards to a collecting channel. The cake formed on the outside of the filtering cylinders is continuously flung off as it is carried round, as a result of the slower rotation of these cylinders around their axes. To regulate the feed the central cylinder is made double walled and the inner wall has a slight vertical movement, so that the feed holes can register or not.—B. M. V.

**Clarifying liquids containing finely divided matter in suspension; Apparatus for —.** W. McD. Mackey. E.P. 154,766, 21.10.19.

A settling apparatus for, e.g., sewage effluent, consists of a tank with vertical walls and sloping curved or flat floor and roof. The feed is at the bottom of the slope, the material having to pass under a certain baffle and through the already settled matter, which acts as a filter for the fresh material; the outlet for clear liquid is at the top of the slope and for settled mud through a controlled spigot or pipe near the bottom of the slope. To make the liquid flow in parallel streams an obstruction may be placed at both the feed and outlet ends, consisting of plates either with a serrated edge or perforated.—B. M. V.

**Presses; Filtering or dewatering — [for peat etc.].** A. Ten Bosch. E.P. 154,817, 19.1.20.

A PRESS for such material as peat consists of a cylinder formed of a number of annular sections bolted together with distance pieces in between so that openings for expressed fluids are left at all the joints, the cross-section of the annular pieces being so shaped that the area for flow of expressed liquid increases rapidly outwards. The cylinder head and piston are so shaped (e.g., hemispherical) that the parts which extend into the cylinder are of gradually decreasing diameter, their greatest diameter being a little less than that of the cylinder. The head and piston may also be built of sections, so as to leave annular openings communicating with their hollow interiors for exit of liquid. The cylinder may be enclosed in a steam jacket, in which case the annular openings in the cylinder will serve both for access of superheated steam to the charge before pressing and for discharge of liquid during pressing.—B. M. V.

**Drying vegetable and like substances; Apparatus for —.** O. Zimmermann and H. Weyel. E.P. 154,998, 5.9.19.

THE material to be dried is fed continuously through two narrow shafts, between and beside which are arranged chambers in which are means for heating the drying fluid. The walls of the shafts are preferably perforated, and the drying medium passes through transversely. The drying medium is passed when hottest over the wettest material in the top of the shafts. In the heating chambers the drying fluid (air) passes over steam coils or the like. Hot furnace gases may be used as drying medium, in which case the temperature may be regulated by addition of air to the circuit, an arrangement of perforated pipes and boxes being used to effect thorough mixing. The shafts may be divided about midway by flaps so that the drying of the material may be effected in two stages.—B. M. V.

**Separating a volatile liquid from solution in a relatively non-volatile liquid; Apparatus for —.** W. K. Lewis and W. Green. E.P. 155,511, 31.1.19.

THE apparatus is intended, more particularly, for the separation of a volatile solvent, e.g., benzol or gasoline, from a non-volatile absorbent, e.g., heavy hydrocarbon oil (cf. E.P. 152,374; J., 1920, 773 A). The mixture is fed to the top of a still, down which it passes in a zig-zag course over trays which are each heated by steam or other means, the clean heavy oil is removed from the bottom of the still, and the volatile solvent (containing some heavy oil) passes out at the top of the still. Live steam may be admitted to the bottom of the still and a vacuum may be maintained. The vapours from the still pass to a first condenser, where any entrained heavy oil is condensed, and then on through a reheater, to which a small quantity of dry air is admitted to improve the vacuum by preventing condensation. The mixture passes to a first vacuum pump and then to a second condenser for clean volatile solvent. Any vapour remaining uncondensed passes on to a second vacuum pump and a third condenser.—B. M. V.

**Mixing materials; Machine for —.** T. J. Sturtevant, Assr. to Sturtevant Mill Co. U.S.P. 1,360,468, 30.11.20. Appl., 24.4.18.

MATERIAL is fed into a horizontal rotating drum having a set of inclined vanes attached to each end, the two sets being inclined in opposite directions to direct material towards the centre. A series of lifting buckets are provided around the periphery at the central zone, and the material is delivered by them on to a fixed inclined shoot, which deflects it towards one or other of the two ends of the drum.

—W. F. F.

*Mixing apparatus for liquids; Automatic* — O. Ritschel. G.P. 323,415, 24.10.18.

AN apparatus for mixing a small quantity of a liquid with a large amount of another different liquid while the latter is under pressure, consists of a container for the former liquid supporting and connected by two tubes with a pressure pipe through which the second liquid flows. One of the tubes allows free entrance of the liquid under pressure into the container, whilst through the other passes a capillary tube connecting the bottom of the container with the pressure pipe. Between the inlets of these tubes into the pressure pipe a regulating valve is arranged, whereby a pressure difference may be set up between the two connecting tubes so that the liquid under pressure is partly forced into the container on one side of the valve and, in its turn, forces an even, regulated stream of the liquid in the container through the capillary tube into the pressure pipe beyond the valve.—A. R. P.

*Gases; Method of generating — and treating solutions therewith.* H. R. Hanley. U.S.P. 1,360,524, 30.11.20. Appl., 15.4.19.

GAS is passed into the liquid at a given level, and the partly spent gas is collected above the liquid surface and re-introduced into the liquid at a lower level.—W. F. F.

*Condenser and method of condensation.* N. H. Hiller. U.S.P. 1,360,748, 30.11.20. Appl., 9.7.15.

THE gas flows upwards through a series of superposed horizontal tubes connected in series, and is cooled and partly liquefied by a surrounding cooling medium. Liquefied gas is drawn off, and a proportion is passed through a conduit, having means for preventing a back flow of gas, to the lower part of the condenser.—W. F. F.

*Filter.* J. P. Probst. U.S.P. 1,361,493, 7.12.20. Appl., 17.3.19.

A VERTICAL cylindrical casing containing filtering material has its vertical walls perforated near the upper and lower ends. The two perforated zones are surrounded by annular chambers, and liquid is admitted to the filter through the lower chamber and discharged through the upper chamber. Contact of the filtering material with the perforations at the upper discharge end of the casing is prevented by a number of closely-spaced vertical strips within the casing over the perforated zone.—W. F. F.

*Evaporation; System of* — W. L. De Baufre. U.S.P. 1,361,834, 14.12.20. Appl., 16.9.16.

AN evaporating chamber and a condensing chamber are connected by a number of ejectors which enable vapour to be withdrawn from the evaporator, compressed, and discharged under pressure into the condenser. The number of ejectors may be varied.—A. J. H.

*Evaporator and condenser.* F. H. Schubert, Assr. to Brown-Ferrier Co. U.S.P. 1,361,910, 14.12.20. Appl., 25.3.19.

A COMBINED evaporator and condenser comprises a single vertical cylindrical casting open at the top, which projects into an annular closed chamber of larger diameter containing the liquid and forming part of the same casting. The lower part of the casting contains vertical condensing tubes fixed in tube-plates at both ends, and a collecting vessel is attached at the bottom.—W. F. F.

*Evaporator.* A. Vincik and F. Turek. G.P. 321,378, 6.3.18. Conv., 8.1.17.

THE apparatus consists of a series of heating chambers arranged side by side in a common vessel, each chamber being separated from the next and

from the enclosing vessel by walls, so that the liquid passes over the whole heating surface of each chamber in succession.—A. R. P.

*Interactions between gas and a liquid; Process of producing* — P. W. Webster and V. K. Boynton, Assrs. to Perry and Webster, Inc. U.S.P. 1,361,940, 14.12.20. Appl., 20.11.19.

A GAS is passed into a liquid in such a finely-divided state as to avoid coalescence of its minute bubbles, thereby removing a portion of the liquid.—B. M. V.

*Cooling liquids; Apparatus for* — Maschinenbau A.-G. Balcke. G.P. 323,709, 19.5.18.

THE cooling surfaces are formed of elastic corrugated sheet metal, concave to the apparatus for spraying the salt solution, so that the salt is deposited on this surface in a bulky mass which, on account of the flexibility and nature of the surface, can readily be detached.—A. R. P.

*Roasting in muffle-furnace; Process of* — H. Frischeber. G.P. 325,073, 18.8.17.

IN cases where material to be roasted cannot be introduced as a counter-current to the heating medium, it may be conveyed into the furnace through the gas or steam exhaust pipe. By this means it undergoes a preliminary heating, and caking in the furnace is prevented.—W. J. W.

*Furnace.* W. G. Zetsche. Reissue 15,009, 14.12.20, of U.S.P. 1,322,214, 18.11.19. Appl., 15.3.20.

SEE J., 1920, 1 A.

*Refrigerating-gas and process of making the same.* A. G. Crawford, Assr. to H. W. Seaman. Reissue 14,955, 12.10.20, of U.S.P. 1,325,666, 23.12.19. Appl., 29.7.20.

SEE J., 1920, 144 A.

*Centrifugal separation.* W. Mauss. U.S.P. 1,355,559, 12.10.20. Appl., 31.10.18.

SEE E.P. 133,448 of 1918; J., 1919, 886 A.

*Evaporating or concentrating liquids; Apparatus for* — E. Shaw. U.S.P. 1,355,702, 12.10.20. Appl., 5.5.19.

SEE E.P. 136,651 of 1918; J., 1920, 144 A.

*Evaporators; Weir-overflow for* — W. H. Ripley, Assr. to The Grisco-Russell Co. U.S.P. 1,361,905, 14.12.20. Appl., 17.5.17.

SEE E.P. 146,730 of 1919; J., 1920, 589 A.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal; "Melting-point" of* — G. Charpy and J. Durand. Comptes rend., 1920, 171, 1358—1360.

IF finely powdered coal, moistened, moulded into a cylinder, and dried at 105° C., is slowly heated in a sealed tube under pressure of dry hydrogen, the cylinder first contracts and then gradually expands again and finally a second contraction characteristic of coking occurs. At the temperature corresponding to the maximum of the first contraction, the coal was noticed to soften and fill up the tube. Eight specimens of coal were examined, and of these a sample of anthracite did not "melt," another sample did not "melt" below 600° C., whilst the others "melted" at temperatures varying between 395° and 550° C. This "melting-point" is independent of the content of volatile matter, but is dependent on the state of oxidation, as the samples when first oxidised at 120° C. did not "melt" on heating as described above.—W. G.

*Peat; Carbonisation of* —. Müller. J. Gasbeleucht., 1920, 63, 817—820.

To be of use for carbonisation peat must not contain more than 25% of moisture or 5% of ash. Such peat yields 30% of gas, 5% of tar, and 0.25% of ammonia at a retort temperature of 1100° C. The drier the peat the less time is required for carbonisation, the period being usually 4—6 hrs. The coke obtained is equivalent to the best gas coke in calorific value, and the coke dust can be advantageously briquetted. The tar is lighter than coal tar and is separated from water with difficulty. It is more valuable than coal tar, giving a higher yield of oils. Large quantities of very dilute ammonia liquor are obtained, the total content of ammonia being large. The gas is heavier than coal gas, but may be purified in the same manner. The carbon dioxide content of the gas is high, being usually about 12%. The calorific value is not less than 4000 cal. In the tests described 6 horizontal retorts were filled with peat and 3 with coal, the coke from 2 of the latter serving for heating the retorts.—W. P.

*Lignite and wood; Carbonisation of* —. R. Geipert. J. Gasbeleucht., 1920, 63, 792—794. (Cf. J., 1920, 180 A.)

The costs and efficiencies of lignite and wood carbonisation processes are compared with those of the carbonisation of coal. Lignite and wood can only be regarded as very poor substitutes for gas coal. The coke produced is poor in quality, and in any case is insufficient in quantity even for heating the retorts. The ammonia liquor from lignite is too dilute to be economically treated for recovery of ammonia.—W. P.

*Coke-oven gases; Composition of some* —. P. Lebeau and A. Damiens. Comptes rend., 1920, 171, 1385—1386.

Four specimens of coke-oven gases were analysed and the following ranges in composition were found:—O<sub>2</sub>, 0—0.69%; CO, 6.41—7.39%; H<sub>2</sub>, 33.30—44.00%; N<sub>2</sub>, 8.06—20.37%; CO<sub>2</sub>, 2.79—4.35%; CH<sub>4</sub>, 23.57—41.34%; C<sub>2</sub>H<sub>6</sub>, 0.43—1.64%; C<sub>2</sub>H<sub>4</sub>, 0.02—0.22%; acetylene hydrocarbons 0.06—0.09%; C<sub>3</sub>H<sub>8</sub>, 1.09—3.68%; propylene and homologues 0.08—0.11%; not determined (moisture, benzene, etc.) 0.36—0.56%. The approximate temperature of the furnaces at the time of taking the samples was 950°—1000° C. These gases differ in composition from coal gas in that they contain less hydrogen, much more nitrogen, and little or no benzene.—W. G.

*Inflammable gases and vapours; Effect of oxygen on the limits of inflammability of* —. E. Terres. J. Gasbeleucht., 1920, 63, 785—792, 805—811, 820—825, 836—840.

The limits of inflammability of carbon monoxide, hydrogen, water-gas, coal gas, methane, ethane, acetylene, ethylene, benzene, and benzene have been determined in air, in pure oxygen, and in atmospheres containing more oxygen than air. In all cases the lower limit differed only slightly in air and in oxygen, but the upper limit was always very much higher in pure oxygen. (Cf. J.C.S., Feb.)—W. P.

*Hydrogen and inert gas [helium]; Inflammability of jets of* —. P. G. Ledig. J. Ind. Eug. Chem., 1920, 12, 1098—1100.

Under favourable conditions a jet of helium containing more than 14% of hydrogen can be ignited in air, but 18—20% of hydrogen may be mixed with helium without producing a mixture which will burn with a persistent flame when issuing from an orifice under the conditions prevailing in balloon practice. More than 20% of hydrogen in a hydro-

gen-helium mixture cannot be used without sacrificing safety from fire.—W. P. S.

*Petroleum refining vapour systems; Prevention of corrosion in* —. R. R. Mathews and P. A. Crosby. Chem. and Met. Eng., 1920, 23, 1122—1123.

To prevent corrosion of the fractionating system by hydrochloric acid generated in the distillation of petroleum containing brine, anhydrous ammonia is added as a neutralising agent. The amount is carefully regulated so as to ensure that the water from the last dephlegmator gives an alkaline reaction. The average amount of iron dissolved per day was 5.18 kg. without, and 2.18 kg. with, ammonia treatment. In addition, the period before tubes had to be replaced in the tubular condenser was extended from 100 to 280 days. Deposits of ammonium chloride must be periodically removed. The substitution of soda ash for ammonia proved unsatisfactory, but aqueous ammonia may be used instead of anhydrous ammonia.—W. J. W.

*Explosion limits.* Jorissen. See VII.

*Gas analysis.* Tour. See XXIII.

#### PATENTS.

*Peat, brown coal or the like; Method of treating — and apparatus therefor.* T. A. Goskar and G. E. Thomas. E.P. 155,012, 8.9.19.

PEAT is mixed with a gritty material such as powdered coal and then compressed, whereby the cells of the peat are broken and the water content liberated. The peat may then be distilled to recover oils etc., and the resulting coke may be crushed and used as the gritty material.—W. F. F.

*Coke oven; Regenerative* —. L. Wilputte, Assr. to A. A. Wilputte. U.S.P. (A) 1,360,609 and (B) 1,360,610, 30.11.20. Appl., 18.9 and 30.9.18.

(A) A SERIES of parallel horizontal coking chambers are heated by vertical flues in the dividing walls. The walls are connected in pairs at their upper ends and gas is supplied alternately to each of the flues of each pair at the bottom. Each flue is connected at the bottom with a separate regenerator. (B) A number of regenerators are arranged side by side with a longitudinal brickwork flue of square cross section running below them, and connected with each by a vertical port. The flue contains a removable metal pipe of circular cross section having openings which register with the ports. The pipe is provided with projecting cam surfaces which occupy the corner of the brickwork flue when the pipe is being inserted, but which engage with the bottom of the flue and lift the pipe against the top of the flue when the pipe is rotated.—W. F. F.

*[Carbonising] oven with regenerator.* C. Berthelot. U.S.P. 1,361,671, 7.12.20. Appl., 15.8.19.

An oven is composed of adjacent carbonising chambers, heated by vertical flues between them. There is a regenerator below each chamber, and a single longitudinal horizontal flue above the vertical flues, and means are provided for connecting one regenerator with one half of the vertical flues between two chambers, and these flues with the longitudinal flue. Means are also provided for connecting the same longitudinal flue with the other half of the vertical flues, and these with the other regenerator below the adjacent chamber.—W. F. F.

*Water-gas producers.* F. G. C. Rincker. E.P. 139,451, 16.9.19. Conv., 24.2.19.

A PAIR of generators working reversibly and connected together are used for the gasification of coal, lignite, peat, wood, etc. Each has an exhaust connexion to the air. In one water-gas is gener-

ated from coke by alternately blowing with air and steam. During the air blast the gas is passed into the atmosphere, but during the steam blast the hot water-gas is passed through the other generator in which its heat effects the distillation of a fresh charge of fuel. When the latter is coked the two generators are reversed. The heat usually lost in the incandescent coke is thus saved.—C. I.

*Water-gas; Generator for producing* —. E. Dolensky, Assr. to The Chemical Foundation, Inc. U.S.P. 1,362,089, 14.12.20. Appl., 11.1.16.

A WATER-GAS generator is connected with a purifier having a water receptacle at the bottom partly open to the atmosphere. A compressed air pipe is connected with the gas pipe between the generator and the purifier, but air is prevented from passing directly into the purifier by a relief branch from the air pipe dipping into the part of the water receptacle open to the atmosphere.—C. I.

*Gas producer.* T. Clouston. U.S.P. 1,362,559, 14.12.20. Appl., 23.8.19.

FINELY-DIVIDED carbonaceous fuel, hot air, and steam are injected axially into one end of a mixing chamber which extends through the gas generating chamber from one end nearly to the other, the inner end being open. The gas generated passes out through a number of short transverse slots in the generating chamber to the delivery pipe. The generating chamber is enclosed in a jacket containing refractory material.—W. F. F.

*Ethylene; Extraction of — from gaseous mixtures [and its recovery as alcohol].* W. Traube. E.P. 147,543, 4.6.20. Conv., 14.7.19.

Gas containing ethylene, e.g., dry coke oven gas, freed from benzene, is sprayed with 90–100% chlorosulphonic acid with cooling. The resulting liquid containing 90–100% of the ethyl ester of the chlorosulphonic acid is decomposed with its own volume of water with formation of alcohol and hydrochloric and sulphuric acids.—C. I.

*Gas; Method and apparatus for cooling* —. A. W. Warner, Assr. to The American Gas Co. of New Jersey. U.S.P. 1,362,207, 4.12.20. Appl., 2.11.18.

Gas from each retort of a series passes through a shower of cooling liquid and into a standpipe which also contains cooling liquid. The gas from all the standpipes passes into a common cooling main where it is further cooled by a shower of cooling liquid.—W. F. F.

*Paraffin wax; Treatment of — for the production of oxygenated compounds [fatty acids etc.].* A. Schmidt. E.P. 142,507, 30.4.20. Conv., 15.1.16.

Air or oxygen is blown through paraffin wax at 100°–120° C. until the colour changes to a deep yellow, i.e., for about 50 hrs. The oxygenated compounds such as fatty acids, oleic acids, and small quantities of aldehydes, alcohols, and the like, are separated from undecomposed paraffin wax, e.g., by distillation. The presence of a catalyst, such as mercury oxide or previously treated wax, increases the rapidity of the reaction.—J. A. C.

*Gasoline; Manufacture of* —. F. E. Wellman. U.S.P. 1,362,160, 14.12.20. Appl., 24.6.19.

PLANT for the manufacture of gasoline comprises a cracking chamber, a discharge pipe provided with controlling valve, a supplemental elevated vapour chamber from which heavy liquid fractions drain back into the cracking chamber, and a condenser connected by a pipe with an elevated part of the vapour chamber. The capacity of the condenser is large relatively to the delivery capacity of the con-

necting pipe at high initial pressure, and the capacity of the pipe is small relative to that of the vapour chamber, so that pressure is maintained in the latter and the vapours expand on passing into the condenser.—J. S. G. T.

*Lubricating oil; Production of — from lignite producer-gas tar oil.* Badische Anilin und Soda-Fahr. G.P. 310,075, 15.4.17.

THE oil obtained by distilling the dehydrated tar is freed from paraffin and heated under pressure to 250°–400° C. Low-boiling constituents are separated from the product by distillation, and are suitable for use in Diesel engines.—L. A. C.

*Lubricating oils; Production of — from coal tar oils.* H. W. Klever. G.P. (A) 310,634, 22.5.18 (Addn. to 301,774–7; J., 1920, 327 A, 441 A), and (B) 310,713, 22.5.18.

(A) A LARGER quantity of oil is allowed to distil during the heating process, and the distillate is returned to the bulk of the oil when the heating is complete. (B) The oil is heated in the presence of a solvent, e.g., oil from naphthalene fractions which is separated afterwards by distillation.—L. A. C.

*Lubricating greases; Manufacture of* —. Rütgerswerke A.-G. G.P. 326,271, 23.2.18.

LUBRICATING oils are heated with tar acids, or tar oil fractions of high acidity, to which lime has been added.—W. J. W.

*Sulpho-acids and alkylsulphuric acids from acid resins [from refining mineral oils and tar oils]. Process for obtaining the salts of* —. Sudfeldt und Co. E.P. 148,579, 10.7.20. Conv., 14.3.19.

THE acid resin obtained, e.g., from the refining of oil with fuming sulphuric acid, is dissolved or emulsified in water or an alkaline solution and subjected to fractional precipitation by the addition of sodium or calcium chloride. Sufficient salt is first added to precipitate sulphonates and hydrocarbons together with only a small portion of the sulphonic acids, and, after separation of the precipitate, the bulk of the sulphonic acids is thrown down by the further addition of salt. The salts of the alkylsulphuric acids and sulphonic acids so precipitated may be purified by solution in water and further fractional precipitation or by treatment with alcohol or with oxidising agents, and may be used as a substitute for asphalt.—L. A. C.

*Powdered fuel; Means for feeding — to furnaces.* L. C. Harvey. E.P. 155,389, 24.9.19.

*Gas making retorts, producers, or the like. Charging of inclined or vertical* —. R. and J. Dempster, Ltd., and G. F. H. Beard. E.P. 155,167, 15.4.20.

*Coke or other material resulting from distillation in vertical retorts; Retaining device for — Apparatus for discharging material of the base of vertical continuous distillation retorts.* J. Pieters. E.P. 155,750–1, 27.2.20.

See also pages (A) 70, *Screening coke etc.* (E.P. 154,376); 71, *Presses for peat* (E.P. 154,817); 80, *Ammonia* (E.P. 155,313); 81, *Hydrogen sulphide from gases* (G.P. 325,966); 92, *Refuse from tanneries* (E.P. 154,961); 103, *Gas analysis apparatus* (E.P. 153,817 and U.S.P. 1,351,129).

## IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Cool; Low-temperature distillation of* —. N. Young. E.P. 134,523, 15.10.19. Conv., 1.11.18.

COAL is continuously distilled at 500°–550° C. in an inclined rotating retort, which is sealed at both ends against the admission of air. The retort is



reated by an external furnace, or by passing hot or superheated producer gas through the charge, or these methods may be combined. The charge is disintegrated during rotation by means of iron rushing balls within the retort. In a modification, two concentric drums are used, both being charged with coal. The coal in the outer drum is partly or completely gasified by admitting air, and the coal in the inner drum is distilled.—W. F. F.

*Mercury vapour lamp; Quartz* — H. George. U.S.P. 1,361,710, 7.12.20. Appl., 11.4.19.

A LAMP, operated by alternating current, comprises a quartz tube connected at each end by a thick-walled, narrow bore tube with a spherical mercury container having an inner spherical bulb communicating with it by a small hole on the side opposite the quartz tube. The leading wires pass into the inner spherical chamber. The quartz tube is filled with an inert gas under pressure which acts as a conductor for the current until enough of the mercury is vaporised.—B. V. S.

*Charcoal; Process of producing decolorising* — R. von Ostrejko, Assr. to Chemical Foundation, Inc. U.S.P. 1,362,064, 14.12.20. Appl., 13.12.16. SEE E.P. 106,089 of 1917; J., 1918, 177 A.

### III.—TAR AND TAR PRODUCTS.

*Lepidine and related bases; Preparation of* — L. A. Mikeska. J. Amer. Chem. Soc., 1920, 42, 2396—2397.

A FAIR yield of pure lepidine (4-methylquinoline) may be obtained by the following process. A mixture of 300 g. of acetone and 300 g. of 40% formaldehyde is cooled in ice and saturated with hydrogen chloride, this process being repeated the next day. On the third day the product is added slowly to a cooled mixture of 300 g. of aniline and 600 g. of hydrochloric acid (sp. gr. 1.2) and the mixture heated on a water-bath under a condenser for 3 hrs. After cooling, the mixture is extracted with ether, the extract is dried, the ether distilled off, and the residue distilled under reduced pressure. The distillate is heated on a water-bath for  $\frac{1}{2}$  hr. with its own weight of acetic anhydride and then poured into water and filtered. The filtrate is neutralised and the lepidine recovered by extraction. If the aniline is replaced by *p*-toluidine, *p*-tolulepidine (4,6-dimethylquinoline) is obtained and if *p*-phenetidine is used 6-ethoxy-4-methylquinoline is the product.—W. G.

#### PATENTS.

*Acid resins.* E.P. 148,579. See II A.

*Lubricating oils.* G.P. 310,075, 310,634, and 310,713. See II A.

*Lubricating greases.* G.P. 326,271. See II A.

### IV.—COLOURING MATTERS AND DYES.

*Isocyanine dyes from lepidine and its homologues.* E. Q. Adams and H. L. Haller. J. Amer. Chem. Soc., 1920, 42, 2389—2391.

THE quaternary additive compounds of pure lepidine or its homologues when treated with alcoholic alkalis in hot, concentrated solution give dyes of the isocyanine type, similar to, but not identical with, those given by the corresponding derivatives of quinaldine. Isocyanines of this type have been prepared from lepidine methiodide and ethiodide and from tolulepidine methiodide, ethiodide, and methonitrate. The isocyanine from lepidine ethio-

dide is probably isomeric with that from quinaldine ethiodide and resembles it in photosensitising properties. (Cf. J.C.S., Jan.)—W. G.

*Photosensitising dyes; Synthesis of* — II. Dicyanine A. L. A. Mikeska, H. L. Haller, and E. Q. Adams. J. Amer. Chem. Soc., 1920, 42, 2392—2394.

WHEN *p*-phenetidine is condensed with paraldehyde and acetone, the product heated with acetic anhydride and then diazotised, and the diazo compound destroyed by heating the solution on a water-bath, 6-ethoxy-2,4-dimethylquinoline, b.p. 314°—316° C. is obtained. If the ethiodide of this base is converted into the ethonitrate and then treated with sodium ethoxide in absolute alcohol, air being bubbled through the mixture, grass-green crystals of Dicyanine A nitrate are obtained, whilst if the ethiodide is treated directly with sodium methoxide in methyl alcohol, Dicyanine A iodide is obtained.—W. G.

*Kryptocyanines. A new series of photosensitising dyes.* E. Q. Adams and H. L. Haller. J. Amer. Chem. Soc., 1920, 42, 2661—2663.

WHEN lepidine ethiodide is dissolved in boiling alcohol and slowly treated with a solution of sodium ethoxide and formalin, air being excluded, a purple-black dye of a new class designated "Kryptocyanines," and specified as "K III," is formed. It has an absorption-maximum near 7000 Å. units and a sensitisation-maximum near 7400 Å. (Cf. J.C.S., Feb.)—J. C. W.

*Azo dyes.* Jacobs and Heidelberg. See XX.

*Mercury derivatives of phthaleins.* White. See XX.

#### PATENTS.

*Disazo colouring matters; Manufacture of* — Brotherton and Co., Ltd., and R. W. Merriman. E.P. 155,410, 11.10.19.

DISAZO dyestuffs which can be applied directly as acid dyes to cotton or wool, giving pink to red shades, or may be after-chromed on wool, giving red shades fast to milling, are obtained by combining *p*-diazobenzencarbazosulphonic acid with a 2-naphthylaminesulphonic acid, such as 2-naphthylamine-5- or -6-mono- or -3,6-disulphonic acid. —L. A. C.

*Nitro compounds, nitroso and azo compounds; Reduction of substituted* — T. S. Moore. E.P. 155,319, 14.7.19.

SEE U.S.P. 1,358,324 of 1920; J., 1921, 6 A.

*Dye base; Process for preparing a* — A. M. Hart. U.S.P. 1,362,879, 21.12.20. Appl., 31.7.18. SEE E.P. 120,588 of 1917; J., 1919, 6 A.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Wood and wood pulp infection and decay.* O. Kress. Pulp and Paper Mag., 1920, 18, 1225—1229.

IN a study of the various moulds and wood-destroying fungi commonly found on pulp or pulp wood, more than seventy different kinds of organisms were isolated. The chemical changes involved in decay of the four chief pulp woods (spruce, hemlock, balsam, and aspen) are considered, and complete analyses of sound and infected woods are reported. The progress of decay is indicated most distinctly by decrease in the content of  $\alpha$ - or stable cellulose and increase in the content of  $\beta$ - or unstable cellulose. Papers made under similar conditions from infected pulps, or pulps prepared from woods in varying degrees of decay, show decreasing strength

tests compared with paper made from sound pulps, especially in those tests which measure the tenacity and the brittleness of the pulp. If pulp is prepared from infected wood it shows a remarkable decrease in fibre length, it is much darker in colour, and it produces a paper with a decidedly reduced strength. It will also require more sizing than sound pulp or pulp produced from sound wood.

—J. C. K.

*Cellulose; Conversion of — into glucose [dextrose]. Constitution of polysaccharides. II.* J. C. Irvine and C. W. Soutar. Chem. Soc. Trans., 1920, 117, 1489—1500.

In previous researches on the hydrolysis of cellulose the yields of dextrose which have been claimed have not been based on the quantity of the sugar or of a characteristic derivative actually isolated, and the statements made are unreliable. With the object of ascertaining whether cellulose is really essentially a polyglucose anhydride, yielding the theoretical quantity of dextrose on hydrolysis, a normal cotton cellulose was hydrolysed by means of acetic anhydride and sulphuric acid. The product was poured into water and the precipitate of polysaccharide acetates simultaneously hydrolysed and converted into methylglucoside by heating in an autoclave with 0.5% methyl-alcoholic hydrogen chloride. About 25% of the material resisted hydrolysis by this method and was hydrolysed with dilute aqueous hydrochloric acid and the product again brought into reaction with acid methyl alcohol. The water-soluble products first obtained were also converted into methylglucoside, and in this way a total yield of pure crystalline methylglucoside was obtained in three distinct stages of progressively increasing difficulty, amounting to 85% of the quantity theoretically possible if cellulose consisted entirely of dextrose residues. Evidence of the non-uniformity of the dextrose linkages in cellulose is thus afforded by the variation in the ease with which the dextrose residues are eliminated from the polysaccharide by acid hydrolysis. The proportion of the cellulose molecule which is most resistant to hydrolysis agrees closely with that corresponding with the maximum yield of cellobiose octa-acetate obtained from cellulose, and this disaccharide apparently represents the most resistant portion of the cellulose molecule.—G. F. M.

*Cellulose; Decomposition of — by aerobic bacteria.* J. Groenewege. Med. Genesck. Lab. Weltevreden, 1920, 163—269.

THERE are two views as to the decomposition of cellulose in nature: one that it is anaerobic and brought about by organisms which are not positively determined; the other that it is aerobic and brought about by two groups of organisms, one group decomposing cellulose directly, the other group being denitrifying organisms. Extensive experiments were carried out from the second point of view, all the cultures being made aerobically. A septic tank liquid and earth were used as the sources of the organisms. The culture medium consisted of tap water to which were added 2% of filter paper, 0.25% of potassium nitrate, and 0.05% of dipotassium phosphate. When this medium was inoculated with either of the substances mentioned and incubated at 37° C. gas was given off, the nitrate was reduced to nitrite and to nitric oxide, and the paper was reduced to a pulp; if at this stage the solution was poured off from the pulp and fresh medium (without filter paper) added, reduction of nitrate again took place, and the pulp was gradually dissolved. The organisms in these cultures were sub-cultivated on agar plates and pure cultures obtained. These were found to fall into three groups: those which effected denitrification but did not attack cellulose; those which did not denitrify but attacked cellulose;

lose; and those which did neither. The first group contained *Bacillus viscosus* and *B. opalescens*, and the second group *B. cellaresolvens*  $\alpha$ ,  $\beta$ , and  $\gamma$ . Cultivations of each member of one group with each member of the other group were made and full details of the results are given. It is concluded that the three varieties of *B. cellaresolvens* attack cellulose, and that the decomposition products serve as food for the denitrifying organisms, *B. opalescens* and *B. viscosus*, the complete process being thus a symbiotic one. The end products of cellulose decomposition are acetic, butyric, and lactic acids, which are oxidised to carbon dioxide and water by the denitrifying organisms. It is suggested that the decomposition of the cellulose may be brought about by an enzyme, cellulase.—J. H. J.

*Paper; Perishing of — in Indian libraries.* J. J. Sudborough and M. M. Mehta. J. Indian Inst. Sci., 1920, 3, 119—226.

PAPER in Indian libraries, especially in Plains stations such as Calcutta, Madras, and Bombay, will not withstand the climate and perishes very rapidly. This is more noticeable with papers prepared from wood pulp, and especially those in which the fibres have been weakened because of the different treatments to which they have been subjected. The addition of arsenic trisulphide as a preservative is useless, because, although it preserves paper from the ravages of insects, it accelerates the perishing of the fibres. Paper used for books which have to be preserved in stations like those of the Indian Plains should be prepared only from strong rag fibres, should have no distinct acidity, and should withstand a severe test in the folding machine. Its contents of rosin and filling materials should be below certain fixed values. The rag fibres should not be weakened by prolonged digestion with alkali or by over-bleaching, and care should be taken to remove the last traces of bleaching agent and of free acid. (Cf. J., 1898, 595.)

—J. C. K.

#### PATENTS.

*Gas-stopping linings for use in aircraft, and media for fixing the same and processes for manufacturing said linings.* V. C. Richmond. E.P. 154,942, 20.12.16 and 20.7.17.

A FILM suitable for ballonet linings consists of a central gas-stopping layer containing gelatin or glue as its chief constituent as regards low permeability to hydrogen, a protective layer containing nitrocellulose, and an attachment layer containing rubber. To prepare the film a mixture of nitrocellulose jelly 200 lb., butyl acetate 40 galls., naphtha 40 galls., and castor oil (to confer flexibility) 8 galls., is sprayed on to a highly-polished surface at 60°—100° F. (16°—38° C.), preferably at 75° F. (24° C.), and allowed to dry. A mixture of gelatin 5½ lb., water 5½ galls., and glycerin 45 oz., is applied as a second layer, and is allowed to dry. The third layer is formed by spraying on a mixture of 32 oz. of a solution containing nitrocellulose jelly 200 lb., butyl acetate 40 galls., naphtha 36 galls., which mixture is diluted to twice its bulk with butyl acetate; 13 oz. of a rubber solution composed of rubber 8.5 pts. and naphtha 91.5 pts. by weight; 6 oz. of naphtha, and 1 oz. of castor oil. After the third layer is dry, the composite film is peeled off, and may be suitably fixed to a fabric by means of a rubber solution. The film has a permeability for hydrogen of approx. ½ l. per sq. m. per 24 hr. both before and after severe crumpling, and its weight is approx. 70 g. per sq. m. A composite film may be made from separate films each containing rubber and dinitrocellulose by use of an adhesive solution prepared from rubber 5.5 pts., naphtha 83.5 pts., and amyl acetate 11 pts.

—A. J. H.

*tains; Process for removing ink and other — from cloth, paper, etc.* A. S. Cushman. U.S.P. 1,361,833, 14.12.20. Appl., 27.8.20.

*MATERIAL stained with ink, grass, etc., is treated with a dilute solution of potassium permanganate, then with a solution of oxalic acid containing hydrogen peroxide, and afterwards washed with water.*—A. J. H.

*Vat or similar fibre; Method of treating —.* W. J. Robinson, Assr. to American Flax Fibre and Linen Corp. U.S.P. 1,355,571, 12.10.20. Appl., 23.4.19.

EE E.P. 141,982 of 1919; J., 1920, 444 A.

*Food-pulp, cellulose, and the like; Press rollers for pressing out water from —.* Aktiebolaget Karlstads Mekaniska Verkstad. E.P. 149,970, 9.8.20. Conv., 16.8.19.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*ngrain dyes on fibres [wool] by means of nitroso derivatives.* Wahl and Guindon. Rev. Gén. Mat. Col., 1920, 24, 179—182.

*WOOL* is dyed full bright shades, ranging from brown through reddish-black to black, by immersion for 1 hr. in a boiling dye-bath which is acidified preferably with acetic acid and contains a nitrosohenol- or nitrosonaphthol-sulphonic acid (2 mols.) and a diamine (1 mol.). Dyeings so obtained are not fast to rubbing and soaping, but are rendered faster, without much change in shade, by after-treatment with dichromate and sulphuric acid. Suitable diamines are diaminodiphenylmethane, enzidine, *p*-phenylenediamine, aminodiphenylamine, and naphthylenediamines.—A. J. H.

### PATENTS.

*Wool washing and like machinery.* T. W. Hawkins. E.P. 155,416, 21.10.19.

*THE* floor of the washing bowl is sloped and along the lowest part is a mechanical scraper or conveyor, whereby the accumulated sludge is led to a tank situated at one end of the bowl. A screw conveyor is so arranged and works at such a speed as to transfer the sludge, but not the scouring liquor, in this receptacle to another tank where it may be suitably treated.—A. J. H.

*Dyeing vats.* A. A. Vallaëys. E.P. 140,764, 16.3.20. Conv., 21.3.19.

*THE* dyeing vat, the gearing which is fixed independent of the vat, and a hood through which vapours are withdrawn are arranged to form a closed system. The vat is divided by a perforated vertical partition near one of the walls, and the smaller division is further divided by a non-perforated plate extending to within 15 cm. of the bottom of the vat. The dyestuff is introduced into the smaller compartment. Inspection of the vat interior is possible by means of two large flap doors. The hood is maintained under slightly reduced pressure, contains a steam-heated coil to prevent condensation of water, and is connected with the remainder of the system by means of a flap valve.—A. J. H.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Nitrogen oxides; Determination of — in nitrogen fixation processes.* G. B. Taylor. Chem. and Met. Eng., 1920, 23, 1112.

A 2-l. bottle is filled with the gas under examination and oxygen is added, if not already present in

sufficient excess, to convert nitric oxide into peroxide. The temperature and pressure of the gas are determined, and 25–50 c.c. of 1.5% hydrogen peroxide solution, prepared by diluting commercial 3% solution with an equal volume of water and neutralising with *N*/10 sodium hydroxide, is then added and shaken with the gas till the red colour disappears. After standing for 5 mins. the mixture is again shaken, and finally titrated with *N*/10 sodium hydroxide, with methyl orange as indicator. The percentage of NO is found from

$$224a/[v \times (b-p) \times 273/760(273+t)],$$

*a* being the c.c. of alkali, *v* the volume of the bottle, *p* the pressure in the bottle, *b* the barometric pressure, and *t* the temperature of the gas. This formula is accurate for concentrations up to 3% NO. If the temperature of the gas is considerably above room temperature a measured volume of water may be added after the agitation with hydrogen peroxide, and the formula then becomes

$$224a/[(v-v')(b-p-w)273/760(273+t)+224a],$$

where *w*=vapour pressure of water at *t*°, and *v'*=volume of peroxide solution plus water.—W. J. W.

*Phosphoric acid; Production of — by smelting phosphate rock in a fuel-fed furnace.* W. H. Waggaman and T. B. Turley. Chem. and Met. Eng., 1920, 23, 1057—1063.

FOR the continuation of their previous large-scale experiments (J., 1920, 595 A), the authors constructed a furnace of semi-commercial size with auxiliary equipment for burning the combustible gases and collecting the phosphoric acid evolved. The plant comprised an oil-fed furnace capable of dealing with a 700 lb. charge, a dust catcher for the gases and fumes, stoves for burning the combustible gases, a Cottrell precipitator, and an air preheater. The material was in the form of egg-shaped and pillow-shaped briquettes. The results of the last two of six runs were recorded. In the first case over 52% of the phosphoric acid was evolved during fusion of the charge, at the rate of about 80.5 lb.  $P_2O_5$  per hr., with a fuel consumption of 9½ galls. of oil, or 8.5 lb.  $P_2O_5$  per gall. The slag obtained after the furnace had cooled contained 7.99–13.63%  $P_2O_5$ ; the volatilisation was therefore 38–65.3% of the original amount in the charge. The run was not completed. In the second case the yield per gall. of fuel during the last 2 hrs. at the maximum temperature (1500°–1600° C.), was 5.56 lb.  $P_2O_5$ . The phosphoric acid in the slag was 0.77%, indicating a volatilisation of 97% of the original acid. The cost per ton of phosphoric acid, assuming a 90% yield by this process, was estimated to be \$49.83 as compared with \$81.25 when produced as acid phosphate (superphosphate) by the sulphuric acid process.—W. J. W.

*Electrolytic diaphragm cells [for production of alkali and chlorine].* H. K. Moore. Amer. Inst. Chem. Eng., June, 1920. Chem. and Met. Eng., 1920, 23, 1011—1015, 1072—1074, 1125—1128.

IN the unsaturated diaphragm cell filled with brine the gradual increase in strength of the caustic soda tends to cause its diffusion into the anode compartment, with consequent loss of carbon from the anodes caused by the action of nascent oxygen produced by the formation and subsequent decomposition of sodium hypochlorite. Diffusion may be checked by keeping the brine at a higher level in the anode compartment, by the addition of water to the caustic soda to lower the temperature and to decrease the concentration, or by causing the caustic soda to flow away as fast as it is formed. Anodes of Acheson graphite are less readily attacked than those of gas carbon. As a measure of the efficiency of cells the rate of flow which represents the volume or weight of caustic solution per ampère-hour per sq. ft. of diaphragm has been adopted. With a high rate of flow current efficiency is low on account of

the large amount of chlorine dissolved in the brine, and as the rate of flow diminishes the current efficiency rises to a maximum and then diminishes. In running a cell plant with the aid of rate of flow charts it is necessary to regulate each cell individually. The voltage of cells rises when the diaphragms are not washed periodically and is also higher with unpurified than with purified brine; a washed diaphragm is superior to a new diaphragm. Increase of temperature of the brine lowers the cell voltage and increases the rate of flow. Decrease of chlorate in the caustic liquor with increase of temperature is probably attributable to the increased activity of nascent hydrogen at the cathode. High efficiency in a cell is sometimes due to a temporary lag in the running-off of the caustic liquor. Small quantities of sodium sulphate in the brine have a deteriorating effect, the carbons being attacked to a greater extent and hence the amount of carbon dioxide in the chlorine increased. The presence of sulphate further increases the amount of chlorate, which constitutes a risk during evaporation of the caustic solution. Numerous graphs and tables of the results are given.—W. J. W.

*Sodium permanganate; Electrolytic preparation of*  
— C. O. Henke and O. W. Brown. *J. Phys. Chem.*, 1920, 24, 608—616.

THE effect of temperature, current density, and concentration of the alkali upon the production of sodium permanganate was studied, using an anode containing 92% of manganese, the impurities being for the most part silicon, iron, and carbon. The anode was prepared by reducing pyrolusite with coke in an electric arc furnace. The cathode was a perforated platinum plate, and the electrolyte a solution of caustic soda containing 10 g. NaOH per litre. The presence of a diaphragm is not necessary if an excess of calcium hydroxide is added to the electrolyte. This addition increases the current efficiency from about 16% to 33%. The calcium hydroxide appears to form a film over the cathode, which acts as a diaphragm. The highest current efficiency was obtained with a current density of about 13 amps. per sq. dm. With a low temperature the current efficiency is increased, i.e., from 3.1% at 60° and 20.1% at 40° to 38.6% at 8° C. As in the case of lead (J., 1920, 603 A), when used as an anode in sodium hydroxide solutions, the discharge potential is higher when the manganese goes into solution with a higher valence, i.e., as permanganate.—J. C. K.

*Phosphates; Volatilisation loss of phosphorus during evaporations of — with sulphuric acid or fusions with pyrosulphate.* W. F. Hillebrand and G. E. F. Lundell. *J. Amer. Chem. Soc.*, 1920, 42, 2809—2815.

No volatilisation losses of phosphorus occur during evaporation of sulphuric acid solutions of phosphates, provided the evaporation is carried on at 150° C. and stopped when fumes appear. Volatilisation losses of phosphorus from sulphuric acid solutions of phosphates may occur during evaporation to complete expulsion of sulphuric acid, during evaporation at high temperatures, such as 200°—260° C., and by unduly prolonged evaporation above 150° C. Evaporations involving only a drop or two of sulphuric acid, such as apply in silica treatments, do not cause measurable losses. Fusion with pyrosulphate in covered crucibles and at dull red heat results in appreciable losses in the case of secondary phosphates, and may result in slight losses in the case of basic phosphates. Orthophosphoric acid and primary and secondary orthophosphates suffer conversion to pyro- and metaphosphoric acid or phosphates when evaporated to dryness with sulphuric acid or fused with pyrosulphate; such "converted" compounds must be subjected to reconversion treat-

ments before precipitation with molybdic acid or magnesia mixture.—J. F. S.

*Adsorption by precipitates [of hydrous aluminium oxide].* H. B. Weiser and E. B. Middleton. *J. Phys. Chem.*, 1920, 24, 630—663.

HYDROUS aluminium oxide adsorbs ions relatively strongly, the adsorption values being approximately one-third larger than those for hydrous ferric oxide. The results obtained for aluminium oxide confirm those obtained previously, viz., that the amount of various ions carried down by the precipitated colloid are not even approximately equivalent, contrary to Freundlich's assumption. The order of adsorption as estimated by direct analysis is not the same as the one deduced from the precipitation values of electrolytes, because the latter do not depend exclusively on the valence and adsorbability of the precipitating ions.—J. C. K.

*Hydrogen peroxide; Properties of pure —.* O. Maass and W. H. Hatcher. *J. Amer. Chem. Soc.*, 1920, 42, 2548—2569.

PURE hydrogen peroxide is prepared as follows: 3% peroxide obtained from barium peroxide is concentrated to 30% by means of a sulphuric acid concentrator (see p. 69 A). It is then distilled at low pressure to remove non-volatile impurities, concentrated to 90% by the sulphuric acid concentrator and pure hydrogen peroxide then separated by fractional freezing. In this way a 50% yield of 100% hydrogen peroxide is obtained. Pure hydrogen peroxide is not very soluble in ether; it dissolves many salts and attacks glass. When sodium is added it explodes violently. It whitens but does not permanently destroy living skin. It is quite stable if kept at 0° C. It has the following physical properties: Freezing point, -1.70° C. sp. gr. of liquid at 0° C., 1.4633; mean coefficient of expansion, -10° to +20° C., 0.00107; sp. gr. of solid, 1.644; sp. heat of liquid, 0.579; latent heat of fusion, 74 cal.; specific heat of solid, 0.470; viscosity at 0° C., 0.0128 (Ostwald);  $n_D^{20}$  = 1.4139 (cf. J.C.S., Feb.)—J. F. S.

*Hydrogen peroxide; Properties of pure —.* O. Maass and O. W. Herzberg. *J. Amer. Chem. Soc.*, 1920, 42, 2569—2570.

THE freezing point curve of mixtures of hydrogen peroxide and water shows that the compound 2H<sub>2</sub>O·H<sub>2</sub>O<sub>2</sub> exists and has m.p. -51° C.—J. F. S.

*Autoxidation [cf phosphorus]; Pressure limit of — considered as a particular case of the inferior limit of explosion.* II. [Explosion limits of acetaldehyde.] W. P. Jorissen. *Rec. Trav. Chim.*, 1920, 39, 715—719.

A COMPARISON of the influence of carbon dioxide or the pressure limit of phosphorescence of phosphorus and on the inferior limits of explosion of hydrogen and methane. In each case the influence of carbon dioxide is greater than that of nitrogen. The limits of explosion of mixtures of acetaldehyde and air were found to be 5.7 and 13.5% by vol., whereas with a mixture of 21% O<sub>2</sub>, 60% CO<sub>2</sub>, and 19% N<sub>2</sub> by vol. in place of air the limits were 8.1% and 11% of acetaldehyde by vol.—W. G.

*Flake graphite; Separation of mineral matter from natural —.* W. C. Ratliff and J. D. Davis. *Chem. and Met. Eng.*, 1920, 23, 1027—1028.

REMOVAL of mineral matter from flake graphite is specially desirable in the case of graphite dust in order to provide a marketable product for the manufacture of stove polishes, graphite paints and lubricants, and electrodes. If the finely ground graphite is agitated with paraffin oil and water, it agglomerates with the oil, and the mineral matter suspended in the water may then be separated. Lighter and more volatile liquids which are im-

iscible with water, such as benzene, toluene, and carbon tetrachloride, are more effective than oil and more readily recoverable, and of these carbon tetrachloride gives the cleanest separation. The amounts of mineral matter retained after treatment by Alabama graphite containing originally 1.68%, were 4.28% with oil, and 3.30–3.44% with the other liquids, while with Pennsylvania graphite the mineral matter was reduced from 10.03% to 4.3% after oil treatment, and to 5.54–5.71% after treatment with the other liquids. Fineness of grinding has an important bearing on the result; in almost every case a smaller amount of mineral matter was retained by graphite which had been ground for 20 hrs. than by that which had been ground for only 8 hrs. The effect of grinding is not so much to reduce the size of the flakes as to diminish their thickness and so expose the mineral matter between the laminae.—W. J. W.

*Graphite; Method for rapid analysis of* — G. B. Taylor and W. A. Selvig. U.S. Bureau of Mines, Bull. 112, 1920, 43–45.

The following method is used at the Pittsburgh laboratory of the U.S. Bureau of Mines:—If much moisture is present the sample is ground to pass through a 60-mesh sieve. Moisture is determined by heating 1 g. in a weighed platinum or porcelain crucible,  $\frac{1}{2}$  in. deep by  $1\frac{1}{2}$  in. wide, for 1 hr. at 105° C. The crucible is cooled in a desiccator over sulphuric acid and the loss of weight noted. Volatile matter is determined by heating the dry graphite for 3 mins. in a muffle furnace at 800° C., cooling as before, and noting the loss of weight. Ash is determined by heating the non-volatile residue at 800° C. until all the graphite is burned away. Graphitic carbon is taken as the difference between the sum of the percentages of moisture, ash, and volatile matter and 100. Graphitic carbon in an ore is determined by heating 0.2–1.0 g. in a 100 c.c. evaporating dish with 25 c.c. of hydrochloric acid (1:1) on a hot plate for 15 min., filtering the mixture through ignited asbestos and washing the residue with hot water. The filter and residue are transferred to a porcelain or platinum boat, dried on a hot plate, transferred to a combustion tube (containing fused lead chromate to retain any sulphur present), and burned in a stream of oxygen. The carbon dioxide formed is passed into a weighed potash bulb containing 30% potassium hydroxide solution and weighed. Graphitic carbon is determined in a concentrate by placing 0.2–0.5 g. of the sample in a small Erlenmeyer flask, adding about 25 c.c. of ether, corking loosely, and allowing to stand for  $\frac{1}{2}$  hr., shaking at intervals. The mixture is filtered through ignited asbestos, the residue washed with alcohol and distilled water, then treated with acid to remove carbonates, and the determination continued as described for ores.—A. B. S.

*Liquefaction of air; Physical principles of the* — R. Plank. Z. ges. Kälte Ind., 1920, 27, 1–8. Chem. Zentr., 1920, 91, IV., 705.

In the liquefaction of air the cooling due to the Joule-Thomson effect for small differences of pressure has been found to be above  $\frac{1}{2}$  at 200° abs. over  $\frac{1}{2}$  at 175° abs., and about 87% at 150° abs. of that produced by adiabatic expansion. Adiabatic expansion is never fully attained in practice.—W. J. W.

*Hydrogen-helium mixtures.* Ledig. See IIa.

*Equilibrium  $Cl_2 + H_2O \rightleftharpoons HCl + HClO$ .* Clark and Iseley. See XIXa.

#### PATENTS.

*Nitric acid; Manufacture of* — N. C. Hill and H. L. Moyler. U.S.P. 1,362,418, 14.12.20. Appl., 28.4.20.

In an apparatus for the continuous manufacture of

nitric acid, which includes a rotating retort, the alkali nitrate is introduced midway into a flow of hot sulphuric acid, which gradually merges into a flow of molten sulphate. The nitric acid vapours are drawn off at a point near the origin of the sulphuric acid flow.—W. J. W.

*Nitrogen oxides; Process for the catalytic conversion of ammonia into* — Badische Anilin und Soda Fabr. G.P. 301,362, 9.3.15.

The catalyst consists of copper oxide mixed with a subordinate quantity of a compound of lead or manganese containing active oxygen. For example, 5 pts. of granular cupric oxide is soaked in 1 pt. of an aqueous solution of lead or manganese nitrate and the mass heated. By passing a 7% ammonia-air mixture over the catalyst heated to 750° C. a yield of over 90% of nitrogen oxides is obtained.—A. R. P.

*Nitrogen oxides; Process for melting solid* — in the freezing apparatus for condensing nitrous gases. Norsk Hydro-Elektrisk-Kvælstofaktieselskab. G.P. 325,636, 29.8.19. Conv., 14.10.18.

So much nitrogen tetroxide is added that the heat furnished by its condensation is sufficient to melt the solidified nitrogen tetroxide, or, alternatively, nitric oxide is introduced, which combines with the solid nitrogen tetroxide to form the trioxide, which melts at a very low temperature.—A. R. P.

*Nitrogen; Regenerative furnace for combustion of* — Gewerkschaft des Steinkohlenbergwerks "Lothringen," and M. Kelting. G.P. 325,635, 31.12.19.

In a regenerative furnace for the combustion of nitrogen, at each side of the combustion chamber there is a heat regenerator consisting of two or more concentric tubes. One or both of the reacting gases are pre-heated in the regenerator on one side of the combustion chamber, whilst the regenerator on the other side is being heated by the exhaust gases from the furnace.—W. J. W.

*Hydrobromic acid; Process for manufacture of* — from bromine and hydrogen. H. R. Napp. From A. Tschudi. E.P. 154,472, 6.1.20.

BROMINE is vaporised in an electrically-heated quartz vessel, having a vertical quartz tube above it into which the bromine gas passes and comes in contact with hydrogen introduced through a lateral inlet. The mixed gases are ignited by bringing the tube to reaction temperature by means of an electrically heated spiral, after which the heat of reaction suffices to maintain the requisite temperature. To ensure that the gases are being admitted in molecular proportions, a sight glass is fixed in the quartz tube through which the colour of the flame can be observed. The hydrobromic acid is conducted to an absorption system.—W. J. W.

*Ammonia-alum; Production of iron-free* — from aluminium sulphate solutions containing ferric sulphate. R. Gans. E.P. 132,510, 8.9.19. Conv., 21.9.17.

FERRUGINOUS aluminium sulphate solution, which may be obtained by dissolving aluminous rock in sulphuric acid, is treated with an excess of ammonium sulphate dependent on the amount of ferric sulphate present. The solution is then caused to react with a base in such quantities as to convert the ferric sulphate, remaining in the solution after crystallisation of the ammonium alum, into basic ferric sulphate, such crystallisation being conducted under agitation with a view to the formation of small crystals; these are finally washed with ammonium sulphate solution. Alum prepared by this method contains considerably less than 0.1% Fe referred to the alumina content.—W. J. W.

*Blast-furnace slag; Arrangement for blowing air through — in particular for recovering the sulphur as sulphur dioxide.* L. H. Diehl. E.P. 139,173, 16.2.20. Conv., 24.9.17. Addn. to 139,172.

For the process of blowing air through blast-furnace slag described in the original patent, a trough is used consisting of sections of hematite iron in the shape of gutters, with external air chests, the sides of the gutters being extended upwards by slabs. In the sides of the gutter sections and in communication with the air chests are air nozzles which are directed obliquely downwards, by which means the slag is kept violently agitated without the formation of a crust at the bottom, the air chests serving to prevent the gutters melting, whilst at the same time the air becomes heated. Lateral inlets and outlets for the slag are so arranged that by means of an overflow level above them, forming a seal, communication with the outside air is avoided, and the gases can only escape through a gas outlet, which passes round the rear end of a vaulted roof beyond the blast zone, and is thus protected against choking by slag spray. The slag crusts can be scraped out through a door in the front of the trough, and openings in the air chests which can be closed by doors enable the air nozzles to be cleaned from outside.—W. J. W.

*Magnesium hypochlorite; Production of solid basic —.* G. Kereszty and E. Wolf. E.P. 142,081, 2.2.20. Conv., 19.4.19.

To obtain a satisfactory yield of solid basic magnesium hypochlorite by passing chlorine into a suspension of magnesia in water, there should be 5½ mols. of excess magnesium oxide for every mol. of hypochlorite formed. A better yield is obtained at 20° C. than at lower temperatures, there being no appreciable quantity of chlorate formed. If, finally, the reaction mixture is either allowed to stand for a week in the dark, or is heated for 6–8 hrs. gradually to 80° C., practically 100% of the total active chlorine is found in the precipitate. While drying the latter carbon dioxide must be excluded.—C. I.

*Potassium carbonate and sodium carbonate; Process for manufacture of — by wet process.* S. Lamm. E.P. 154,498, 2.3.20.

FINELY ground potassium sulphate and barium sulphide are intimately mixed with granular potassium bicarbonate and added to boiling water in a heated vessel. The nascent potassium sulphide formed by interaction of the first two salts reacts with the bicarbonate without secondary reactions taking place, and gives a product of a high degree of purity. The solution is separated from the precipitate, concentrated, and crystallised. The residual barium sulphate is utilised for regenerating barium sulphide, and the hydrogen sulphide evolved is available for other purposes. Sodium carbonate is manufactured by an analogous method.—W. J. W.

*Potassium bichromate and sodium bichromate; Manufacture of —, and production of normal sulphates of potassium and sodium.* R. L. Datta. E.P. 154,810, 5.1.20.

POTASSIUM chromate liquor is heated and treated with acid potassium sulphate cake, the latter being added until conversion of chromate into bichromate is complete. The solution is filtered, the potassium sulphate separated, and the mother liquor is crystallised. Sodium bichromate may be similarly produced by the interaction of nitre cake with sodium chromate liquors.—W. J. W.

*Ammonia; Production of —.* A. Rollason. E.P. 155,313, 17.6.19.

A PRODUCER, constructed with an outer shell surrounding the producer chamber and provided with a superheater, is charged with pulverised coke, anthracite, or hard coal, the fuel being kept at a uniform height by means of an internal bell arrangement. Air, saturated with steam, is preheated by passing through the superheater and outer shell and forced at slightly above normal pressure into the fuel bed, the temperature of this being kept below 950° C. by introducing steam. The gases leaving the producer, free from oxygen, are cooled to about 500° C. either by steam or by means of a cooling arrangement, and enter a decomposer consisting of a steel or cast iron outer tube lined with firebrick and an inner tube, into which the gases pass and in which they are heated to 550°–600° C. by hot gases which circulate between the two tubes, thus causing the dissociation of the nitrogen molecule into free atoms. The gases are then conducted into the inner tube of a converter which is charged with breeze or coke to which limestone has been added, and is kept at 700°–800° C. by hot circulating gases. The water vapour in the gases is dissociated, the oxygen combines with the carbon, and combination of the free nitrogen and the nascent hydrogen takes place, the gases leaving the converter being cooled to 90° C., and the ammonia recovered. The final gases may be utilised for combustion in furnaces to heat the superheater, decomposer, and converter.—W. J. W.

*Magnesium carbonate; Process of and apparatus for manufacturing [basic] —.* B. B. Grunwald, Assr. to National Magnesia Manufacturing Co. U.S.P. 1,361,324 and 1,361,325, 7.12.20. Appl., 18.3.18.

CALCINED magnesite is hydrated and treated with carbon dioxide under pressure. The resulting product, containing excess of carbon dioxide, is mixed with more calcined magnesite and the mixture heated to precipitate basic magnesium carbonate.—B. V. S.

*Monazite sand; Treating —.* E. L. Knoedler, Assr. to Welsbach Co. U.S.P. 1,361,735, 7.12.20. Appl., 1.5.18.

THE sand is heated with sulphuric acid and the hot mixture poured into a "drowning tank."—B. V. S.

*Titanium hydrates [hydroxides]; Removal of undesired acid substances from —.* C. Jessen, Assr. to Titan Co. A./S. U.S.P. (A) 1,361,866 and (B) 1,361,867, 14.12.20. Appl., 12.10.17.

THE acid is removed by treating the precipitated titanium hydroxide with (A) a compound of a metal forming a neutral insoluble salt with the acid or (B) an alkali which forms a neutral soluble salt.—C. I.

*Sulphur dioxide and metallic sulphates; Manufacture of — from burnt pyrites.* Zellstoff-fabrik Waldhof. G.P. 298,552, 5.10.16.

BURNT pyrites is heated with sulphuric acid or an acid sulphate until the mass becomes dry. The temperature is then raised until all the iron sulphate is decomposed into iron oxide and sulphur dioxide without decomposing other metallic sulphates such as those of zinc or copper. Any unroasted pyrites present in the charge is converted into iron oxide with evolution of a further quantity of sulphur dioxide. The residual mass is extracted with water to remove soluble salts and leave a fairly pure iron oxide. When sodium bisulphate is used in the process sufficient common salt is added to the solution to convert the salts of



the heavy metals into chlorides, and the sodium sulphate present is then removed by crystallisation or by freezing.—A. R. P.

*Sulphur dioxide; Manufacture of liquid* — from sulphur dioxide diluted with other gases. Mann-factures de Prod. Chim. du Nord Etabl. Kuhlmann. G.P. 325,473, 23.11.19.

GASES containing a small percentage of sulphur dioxide, e.g., those obtained from roasting blende or pyrites, are compressed and then agitated with heavy tar oil free from anthracene, which absorbs the sulphur dioxide. The latter is removed from the oil by warming, and liquefied in the usual way. Coal-tar oil of sp. gr. 1 dissolves at atmospheric pressure and ordinary temperatures about a quarter of its weight of sulphur dioxide.—A. R. P.

*Hydrogen sulphide; Process of separating* — from gases. Zschocke-Werke A.-G. G.P. 325,966, 31.7.17.

THE gas is first sprayed with excess of a washing agent, and is then treated with a portion of the liquid in a centrifugal gas-washing apparatus. —W. J. W.

*Nitric acid; Production of* —. K. B. Quinan. U.S.P. 1,355,357, 12.10.20. Appl., 24.2.19.

SEE E.P. 131,029 of 1918; J., 1919, 718 A.

*Salt; Manufacture of table* —. C. Dietz, Assr. to Chemical Foundation, Inc. U.S.P. 1,362,087, 14.12.20. Appl., 14.11.16.

SEE E.P. 18,125 of 1915; J., 1916, 963.

*Tin; Oxidation of* —. [Manufacture of tin oxide.] D. de Ros. U.S.P. 1,362,237, 14.12.20. Appl., 9.8.20.

SEE E.P. 118,664 of 1917; J., 1918, 622 A.

[Calcium] cyanamide; Apparatus for the manufacture of —. A. Duchemin. U.S.P. 1,363,096, 21.12.20. Appl., 15.7.19.

SEE E.P. 142,354 of 1919; J., 1920, 488 A.

## VIII.—GLASS; CERAMICS.

*Glass; Annealing of* —. L. H. Adams and E. D. Williamson. J. Franklin Inst., 1920, 190, 597—631, 835—870.

WHEN judged by an arbitrary standard of annealing—that the maximum optical path-difference in an annealed block shall not exceed  $10\mu$  per cm.—the release of stress in glass during annealing at constant temperature may be represented by

$$\frac{1}{F} - \frac{1}{f} = At$$

where  $F$  is the stress at any time,  $t$ ;  $f$  is the initial stress, and  $A$  is a constant for the particular glass at a particular temperature. The annealing time for optical glass is defined as the time required at a given temperature to reduce the stress from 50 to  $2.5\mu$  per cm. Most of the important operations in glass annealing occur over a temperature range of about  $150^\circ\text{C}$ . which is limited at its upper end by the temperature at which the strain disappears in 2 mins. At temperatures below this annealing range very little permanent stress can be introduced during cooling. The permanent stress acquired during cooling is equal and opposite in sign to the temporary stress when the glass is being annealed, and in order to obtain glass free from internal stress it is necessary to prevent the release of stress during cooling. The best way to do this is to cool the glass slowly through the annealing

range and more rapidly later. Hence, instead of the customary method of annealing in which the glass is cooled slowly from the upper end of the annealing range to room temperature, the authors prefer to maintain the glass for a sufficient time at a temperature at which it requires several hours to release the stresses and then to cool it rather rapidly. With the latter method the chances of failure during cooling are very low. For a plate of ordinary crown glass, 2 cm. thick, the lower end of the annealing range is  $511^\circ\text{C}$ . the cooling period 2.2 hrs., and the total time of heating and cooling is about  $4\frac{1}{2}$  hrs. For optical glass slabs the rate of cooling should never exceed  $10/a^\circ\text{C}$  per min., where  $a$  is half the thickness of the slab in cm. The saving in time afforded by the authors' method is increasingly important with large pieces of glass. Tables of annealing schedules for various optical glasses are given.—A. B. S.

*Iodine and bromine vapours; Permeability of glass to* —. J. B. Firth. Chem. Soc. Trans., 1920, 117, 1602—1603.

IODINE and bromine do not diffuse through a glass partition under ordinary conditions. In an extreme case the diffusion of iodine through a glass bulb 0.21 mm. thick was observed after  $9\frac{1}{2}$  years, during 100 days of which the bulb had been heated to  $360^\circ\text{C}$ . Even under these conditions there was no evidence of the diffusion of bromine.—G. F. M.

*Flake graphite; Refining Alabama* — for crucible use. F. G. Moses. U.S. Bureau of Mines, Bull. 112, 1920, 46—74. (Cf. J., 1919, 633 A.)

INVESTIGATIONS on refining crude graphite concentrates showed that no one refining treatment is equally applicable to all concentrates on account of the different characteristics of the crude graphites and their impurities. When the impurities are free or soft, they can easily be separated by grinding, but hard impurities interlaminated with the graphite are more difficult to separate. An aspirator and pneumatic jig are only useful for removing the coarse, hard and free impurities. An electrostatic separator or pebble mill, followed by flotation, may be used for all Alabama graphite concentrates, but a buhr mill is also essential in "finishing" the graphite and preparing it for the removal of interbedded impurities. The use of this machine presupposes that the impurities are ground much more finely than the graphite and so are separated in the subsequent screening. It should be possible to keep the recovery of graphite coarser than 100-mesh at 70% or more of the total carbon in the original concentrate. If only high-grade graphite is sent to the buhr mill it is not difficult to obtain a final product with over 90% of carbon. Care must be taken not to produce too much fine flake which will pass through a 100-mesh sieve. A loss of only 20% of fine graphite is not objectionable. Coarse grit in the material supplied to the buhr mill is the chief cause of loss through grinding the carbon too fine for use in crucibles, i.e., finer than 100-mesh.—A. B. S.

*Flake graphite [for crucibles]; Tentative specifications for* —. G. D. Dub. U.S. Bureau of Mines, Bull. 112, 1920, 38—41.

TENTATIVE specifications suggested by the Columbus Experiment Station of the U.S. Bureau of Mines for No. 1 flake graphite are as follows:—The graphite carbon content, determined by weighing the residue left after heating the dried sample for 3 mins. at  $800^\circ\text{C}$ ., should not be less than 85%. The cumulative percentage residues on a series of screens should be: 35% on 35 standard mesh, 50% on 65 standard mesh, and 100% on 100 standard mesh. The permissible allowance is not more than: 3% through a 100-mesh screen.—A. B. S.



*Graphite.* (1) Ratliff and Davis. (2) Taylor and Selvig. See VII.

#### PATENTS.

*Glass compositions and articles made therefrom.* Corning Glass Works, Assees. of E. C. Sullivan and W. C. Taylor. E.P. 145,423, 17.6.20. Conv., 26.2.17.

CLAIM is made to a glass containing silica, soda, and at least one oxide of a divalent alkaline-earth, the molecular ratios falling within the following limits: silica 100, alkali oxide 20 to 36, and oxide of divalent alkaline-earth 6 to 36. Magnesia forms at least 40% of the alkaline-earth oxide content. Magnesia glasses have lower coefficients of expansion than lime glasses of equal softness and are suitable for electric lamp bulbs.—H. S. H.

*Brick dryers; Supply of heat to tunnel* —. T. Entwisle. E.P. 154,966, 16.8.19.

IN an improved method of supplying heat to tunnel brick dryers, hot air from the cooling chambers of a continuous kiln is passed through an auxiliary flue to a mixing chamber provided with a cold air inlet and a fan, and thence through ducts below the tunnel dryer and opening up into the latter. The passage of the hot gases is controlled by dampers.—A. B. S.

*Drying machines [for pottery].* T. Allsop and W. W. Sibson. E.P. 154,896, 3.9.19.

A CASING is divided by partitions into a heating compartment, a drying compartment, and an auxiliary compartment. The goods to be dried, e.g., pottery, are moved through the second and third, and the drying medium, such as air, circulates between the first and second compartments. The goods are supported on long carriers, which extend practically across the full width of the casing, space being left only for a pair of conveyor chains which support the carriers and pass very slowly up and down several times over sprockets in the drying chamber, returning through a duct underneath and upwards through the auxiliary chamber, where they are unloaded and charged with fresh goods. The heating chamber may be provided with cross tubes heated by steam, and the air circulated by a fan in the partition between the heating and drying chambers.—B. M. V.

*Tunnel kilns or furnaces.* R. C. Metcalfe. E.P. 155,164, 7.4.20.

A TUNNEL kiln is provided with a single reciprocating truck to transport goods back and forth between a high temperature zone in the kiln and an inlet and outlet disposed laterally at right angles to, and at opposite ends of, the high temperature zone. The ends of the kiln chamber are so shaped that the truck prevents gases in the high temperature zone from passing out at either end of the chamber.—A. B. S.

*[Tunnel] ovens or kilns; Gas-fired — for tiles, pottery, etc.* J. H. Marlow. E.P. 155,190, 30.7.20.

A GAS-FIRED tunnel oven has a number of burners of different diameters arranged to produce a gas flame at each side of the oven, and two combustion chambers at each side of the oven. Each combustion chamber communicates with a series of air pipes so that when the oven has reached a predetermined temperature the air in the pipes can be cut off and replaced by air heated recuperatively in a second series of pipes which supply heated air to the second combustion chamber on each side of the oven. The flames and products of combustion then pass over the auxiliary burners and away to the chimney.—A. B. S.

*Furnace structures [linings].* J. H. Cumpsty. E.P. 155,315, 20.6.19.

A MONOLITHIC furnace lining or other structure is made by arranging suitable metal reinforcement in a mould and filling the latter with a thin cement composed of magnesia and sodium silicate or like binding material, or with a mixture of broken magnesite bricks or magnesite and 10% of sodium silicate. The moulded article is dried and fired and is then ready for use.—A. B. S.

*Pottery ware [; Ornamenting —].* A. P. Morris. E.P. 155,332, 15.8.19.

BLACK Burmese pottery, in which the black coloration is produced by the deposition of carbon from the smoke towards the end of the burning period, may be ornamented by removing the carbon from predetermined areas by oxidation and so revealing the natural colour of the burned clay. The oxidising or decarbonising material may consist of potassium chlorate, potassium nitrate, or the like made into a paste and painted on the ware, which is then re-fired at a low temperature (400° C.) sufficient to effect the oxidation but not to cause the oxidising agent to adhere to the ware. The re-fired ware when cold is freed from any excess of oxidising material, which usually flakes off.—A. B. S.

*Pots; Impts. in and in the manufacture of acid — to be employed in the production of white lead.* H. T. Arrowsmith. E.P. 155,373, 17.9.19.

EACH pot is moulded with two or more projections on its upper edge, and after glazing inside the pots are stacked with their mouths together. The projections prevent the pots from sticking owing to the glaze flowing on to the edges of the pots. Each pair of pots forms a chamber and the glazed interior is thus protected from contamination during firing so that no saggars are necessary.—H. S. H.

*Refractory article and process of making same.* F. J. Tone, Assr. to The Carborundum Co. U.S.P. 1,362,274, 14.12.20. Appl., 24.2.20.

A HIGHLY refractory material consists essentially of a burnt mixture of zirconia and sillimanite.—H. S. H.

*Refractory material and process of making it. Refractory article and process of making same.* O. Hutchins, Assr. to The Carborundum Co. U.S.P. (a) 1,362,316 and (n) 1,362,317, 14.12.20. Appl., 7.1 and 24.3.20.

(A) A HIGHLY refractory material consists essentially of a burnt mixture of zirconia and alumina. (B) A shaped highly refractory article is formed of a burnt mixture containing zirconia and chromite.—H. S. H.

*Glass charges; Method of getting viscous —.* O. M. Tucker and W. A. Reeves. E.P. 131,586, 5.8.19. Conv., 12.8.18.

*Glass; Apparatus for manufacture of articles of — from glass tubing.* W. A. Whatmough. E.P. 155,433, 10.11.19.

## IX.—BUILDING MATERIALS.

#### PATENT.

*Cement; Slow setting, fat and water proof — and method of producing same.* H. D. Baylor. E.P. 155,431, 8.11.19.

SEE U.S.P. 1,323,953 of 1919; J., 1920, 113 A.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Steel; Inclusions and ferrite crystallisation in —.*  
*II. Solubility of inclusions.* E. G. Mahin and E. H. Hartwig. *J. Ind. Eng. Chem.*, 1920, 12, 1090—1095. (Cf. *J.*, 1919, 864 A.)

A CONSIDERABLE number of alloys and special steels were turned into small rods and driven into holes in carbon steels, the specimens being then heated to temperatures above the transformation range for the steel. The section of the slowly cooled piece showed, in nearly every case, ferrite segregation around the insert, but the ferrite ring varied greatly in width. It is suggested that the special element, elements, or compounds present in the insert diffused into the steel and caused, in varying degrees, separation of ferrite from the austenitic solution when the latter was cooling through the transformation range. If this hypothesis is correct, the importance of the uniform distribution of all elements of carbon and alloy steels is evident, as otherwise it is not possible to have uniform carbon distribution and the finished piece cannot be brought into its best condition by any ordinary heat treatment.—W. P. S.

*Mild steel; Inter-crystalline fracture in —.* W. Rosenhain and D. Hanson. *J. Iron and Steel Inst.*, 1920, 102, 23—37.

FOUR cases of inter-crystalline fracture in mild steel are recorded. Some steel wrapper plates from the combustion chamber of a marine boiler cracked between the rivet holes near the edges. The explanation in this case is that near the surface free cementite occurred between the ferrite crystals. The same cause led to a similar failure in a  $\frac{1}{2}$  in. mild steel plate from the drum of a water-tube boiler. Inter-crystalline fracture in steel plates from a high-pressure marine boiler around and between the rivets is ascribed to the application of long-continued stresses over a period of years. A piece of the plate nicked and broken in several hours showed no inter-crystalline weakness. The last case was a mild steel tube which had failed when under no stress at 300° C. in a nitre bath. Cold-drawing stresses had been left in through insufficient annealing which led to failure.—F. C. Th.

*Steel [and other metals]; Effect of nitrogen [ammonia] on —.* O. A. Knight and H. B. Northrup. *Chem. and Met. Eng.*, 1920, 23, 1107—1111.

A PRELIMINARY investigation of the effect of ammonia at 650° C. on various metals and alloys, as indicated by their microstructure, showed that tungsten, chromium-vanadium steels, stellite, nichrome, and Monel metal, are not affected, or only slightly attacked. Wrought iron and low-carbon steels are readily attacked; high-carbon steel is much more resistant, and white cast iron is affected on the surface. The results have only a negative value, as the metals which are not acted on are unsuitable for use in making containers for ammonia either as regards price or physical properties. Nine samples of annealed steel wire were selected for physical examination, six of them being first heated in ammonia gas at 650° C., and of these, three were further heated in hydrogen at 650° C. The treated samples showed a diminished tensile strength and reduced elongation; hydrogen treatment partly restored the normal properties, and entirely removed the irregular "white layer." An investigation was made of the effect of ammonia on all grades of carbon steel. The samples were case-hardened at 950° C., then cut in half in the centre of the longest dimension, the exposed surface varying in carbon from 0.07% near the centre to

1% on the outside. One portion was ground, heated in ammonia at 650° C. for 12 hrs., and furnace-cooled; it was then cut on a plane normal to the previous exposed surface, mounted in an alloy of low fusion point and polished; as no effect was observed, the sample was etched with 4% nitric acid in absolute alcohol. The samples then showed a uniform outside layer which was the same in both the high- and low-carbon areas. Longer etching merely intensifies the corrosive action on the outer three-fifths of the layer, but on the inner two-fifths it develops a crystalline structure of a columnar nature. The base of these crystals forms a sharp demarcation from the pearlitic structure beneath. The outer layer is characterised by extreme hardness and brittleness. In the high-carbon area the pearlitic structure at the base of the columnar crystals appears to be normal; in the low-carbon region, however, the pearlitic layer is succeeded by a layer of apparently normal ferrite grains, and below this the ferrite grains contain a number of "nitrogen needles," proving that nascent nitrogen has a greater penetrating power in low-carbon than in high-carbon steels.—W. J. W.

*Steel, iron, and cast iron; Metallographic methods for determining the nature of the non-metallic inclusions in —.* M. Matveieff. *Rev. Mét.*, 1920, 17, 736—752.

TO detect the presence of ferrous oxide inclusions the polished specimen is heated to dull redness in hydrogen and again polished, when the inclusion, if due to ferrous oxide, disappears. Inclusions of sulphides are best recognised by etching the specimen with a 0.2% solution of oxalic acid, which attacks manganese sulphide and yields a rose-coloured tint with ferrous sulphide. Silicate inclusions are not attacked by either of the above reagents and may be recognised by their dark colour. The distribution of phosphorus in the metal may be examined by etching with a mixture of 100 c.c. of methyl alcohol, 2 c.c. of hydrochloric acid, 1 g. of copper chloride, 4 g. of magnesium chloride, and 18 c.c. of water, when the parts richest in phosphorus remain clearer than the rest of the metal. Details are given of the use of the above reagents in discovering the reasons for the failure of ingots of iron and steel.—A. R. P.

*Steel; Electrometric method for detecting segregation of dissolved impurities in —.* E. G. Mahin and R. E. Brewer. *J. Ind. Eng. Chem.*, 1920, 12, 1095—1098.

A METHOD is described for measuring the electrode potential of a single grain or microscopic point on a metal specimen. When the method is applied to a piece of steel containing a segregated ring of ferrite, produced by heating in contact with aluminium bronze, the average values for the ring are 0.051 volt lower than for ferrite in the unaffected part of the metal. This indicates a different degree of purity for ferrite under the two conditions.—W. P. S.

*Nickel-iron alloys; Constitution of —.* D. and H. E. Hanson. *J. Iron and Steel Inst.*, 1920, 102, 39—64.

THE diagram of the changes which occur below the solidus for pure iron-nickel alloys (maximum carbon content 0.046%) shows that up to 9% Ni this is at room temperatures in solid solution. Between 9 and 32% Ni a eutectoid horizontal occurs at about 350° C., the eutectoid point being at about 25% Ni. The two curves above 30% Ni lie quite close to each other and join at about 700° C. Very long soakings are required to produce the equilibrium structure.—F. C. Th.

*Aluminium; Influence of iron on the mechanical properties of cast —.* L. Guillet and A. Portevin. *Rev. Mét.*, 1920, 17, 753—756.

THE presence of iron in cast aluminium increases

the hardness and diminishes the ductility, metal containing about 4% Fe being so brittle that it cannot be bent without breaking. Figures are given showing the results obtained for aluminium containing 0.7 to 3.9% Fe in the Brinell, repeated impact, and notched bar tests. Photomicrographs show that the alloys have a eutectic structure when the iron content is between 0.7 and 1.7%, while those with higher proportions of iron contain large numbers of needles and star-like aggregates.

—A. R. P.

*Sheet zinc; Occurrence of a foliated appearance on strips of —.* E. H. Schulz and R. Melaun. *Forschungsarb. Geb. Ing., Sonderreihe M.* [1], 23–27. *Chem. Zentr.*, 1920, 91, IV., 622.

On bending and twisting several different kinds of sheet zinc it was noticed that the surface split up into small leaves, giving the material a foliated appearance. While this could be traced partly to segregation of the lead in rolling, it otherwise did not appear to have any connexion with the chemical composition of the metal and was probably due to the method of rolling, the metal having been rolled out in fewer stages than usual, thus giving rise to temperature differences between the outer surfaces and the interior. This produced small longitudinal cracks along the edges of the rolled sheet and on the broken edges of a fracture, and on bending the metal caused the foliated appearance. No explanation, however, could be found for the fact that Rhenish and Belgian zincs behave worse under these conditions than zinc of similar composition produced in Upper Silesia.—A. R. P.

*Forged zinc; Influence of the forging temperature on the tensile properties of —.* E. H. Schulz and R. Fiedler. *Forschungsarb. Geb. Ing., Sonderreihe M.* [1], 27–29. *Chem. Zentr.*, 1920, 91, IV., 622–623.

Tests made on zinc rods (containing 1.3% Pb and a trace of iron) forged down from 120 mm. to 50 mm. diameter at various temperatures showed that the highest tensile strength and elongation were obtained when the forging temperature was 120° C. As this temperature was increased the ultimate stress altered but little, but the elongation and resistance to impact of the resulting bar were considerably decreased, while the metal became coarser grained. Metal containing more iron must be worked at a higher temperature than pure zinc, and if worked just below 120° C. it gives a greater elongation.—A. R. P.

*Forged zinc; Development of a coarsely crystalline structure in — by heating.* E. H. Schulz and O. Zeller. *Forschungsarb. Geb. Ing., Sonderreihe M.* [1], 32–38. *Chem. Zentr.*, 1920, 91, IV., 623.

By heating forged zinc for ½ hr. at gradually rising temperatures from 100° C. upwards and examining the fracture and microscopic structure of polished and etched surfaces, it was established that, at a definite temperature, depending on the forging temperature of the sample, but at least 140° C., a fairly sharply separated zone of larger-grained metal developed round the outer edges of the test-piece. As the temperature was raised this zone gradually increased in extent until at 200° C., or higher, according to the original forging temperature, complete recrystallisation of the metal took place. As the grain of the metal became coarser the tensile strength, and especially the elongation, decreased, the latter very considerably after recrystallisation had set in, but no change was noticed in the Brinell test. Heating zinc to 150°–200° C. has a bad effect on the elongation. The tensile strength is not appreciably altered by heating for ½ hr. at 200° C., but at 300° C. a considerable decrease in strength is produced.—A. R. P.

*Zinc; Behaviour of — in the impact test.* E. H. Schulz and R. Fiedler. *Forschungsarb. Geb. Ing., Sonderreihe M.* [1], 29–31. *Chem. Zentr.*, 1920, 91, IV., 622.

Forged zinc with a tensile strength up to 20 kg. per sq. mm. and an elongation of 30% or more gave results in the impact test with a notched bar varying from 0.55 to 0.75 kg.-m. per sq. cm., and this figure could not be improved upon by preparing the specimen by other methods, by further refining, or by alloying the zinc with other metals. The results obtained with bars with V-shaped notches decreased as the angle of the notch increased, and the broken bars showed a short granular fracture. Bars with a circular notch had a resistance several times as great as those with angular notches, and the fractured surface was very jagged. Tests at various temperatures showed that the resistance of zinc to impact decreased with fall of temperature.

—A. R. P.

*Forged and rolled zinc; Effect of addition of copper and aluminium on mechanical properties of —.* E. H. Schulz and R. Melaun. *Forschungsarb. Geb. Ing., Sonderreihe M.* [1], 38–42. *Chem. Zentr.*, 1920, 91, IV., 621–622.

ADDITION of 2½% Cu to forged zinc increases the tensile strength and hardness of thick bars to an extent depending on the temperature of forging, but does not appreciably improve the ductility or the toughness. Similar results are obtained by the addition of aluminium up to 4.4%, but in this case the metal appears to be more brittle. Thick plates that have been rolled from 30 cm. down to 1 cm. have good tensile strength and ductility combined with a low hardness number if 2% of copper has been alloyed with the metal. Addition of aluminium does not give such good results, it being necessary to add more than 4% Al to get the same effect as with 2% Cu, and even then, for the same tensile strength the elongation is less than that of the copper alloy. According to the particular method of working forged zinc with 2% Cu, the elongation and contraction of area vary from 9 to 30%.—A. R. P.

*Brass; Influence of gases on high-grade —.* T. G. Bamford and W. E. Ballard. *J. Inst. Metals*, 1920, 24, 155–200.

At 300° C. brass absorbs sulphur dioxide, but at higher temperatures, viz., between 950° and 1050° C., and low partial pressures, the gas is eliminated from the metal. Thus the metal to be sound must be poured at such a temperature as to permit it to remain entirely fluid in the mould for a few moments. The conclusions reached regarding the solubility of hydrogen in 70:30 brass are: Between 500° and 900° C. the gas is absorbed in very large quantities depending on the temperature, pressure, and character of the surface. The larger part of the gas absorbed is retained on cooling, but much may be removed by treatment with a neutral gas at 600° C. or above. It is considered that in ordinary foundry practice with a coke fire and natural draught, it is impossible seriously to impair the mechanical properties of the casting by overheating. Cooling to within 40° C. of the liquidus will certainly ruin the mechanical properties of the casting by making it porous. In casting tubes a higher casting temperature is required than when casting solid ingots—at least 1150° C. in the case of 70:29:1 tubes. Exceptionally prolonged heating of the metal in the furnace does not impair the mechanical properties of the brass but results in a high loss of zinc. The casting temperature rather than the furnace treatment is believed to be the important factor in regard to the mechanical properties of the metal.—F. C. Th.

**Beta brass; Behaviour of — when cold-rolled.** F. Johnson. *J. Inst. Metals*, 1920, 24, 301—314.

THE brass examined contained 44.4% Zn. After casting the ingot was annealed for 1 hr. at 650° C. and then reduced in cross-section by 50% without further heat treatment. As a result of the rolling the maximum stress rose from 25.5 tons for the cast and 22.7 tons for the annealed alloy to 38.2 tons per sq. in., the elongation fell from 17% (cast) and 27% (annealed) to 8.6%, and the Brinell hardness (500 kg. load) rose from 85 (cast) and 86 (annealed) to 173. The microstructure of the alloy in the annealed condition consisted of the  $\beta$ -phase exclusively. After the cold rolling the crystals were traversed by numerous lamellae (analogous to the Neumann lamellae observed in meteorites and in ferrite which has been subjected to shock), showing faulting. When the cold-rolled brass was subsequently annealed for 30 mins. at 400° C. it was along these lamellae that the  $\alpha$ -phase made its appearance. The influence of annealing for 30 mins. at different temperatures after cold rolling upon the hardness is shown in the following table:—

Temperature of annealing, °C.	Brinell hardness.		Shore
	500 kilos.	1000 kilos. (magnifier hammer).	
As rolled ..	173 ..	192 ..	55
250 ..	100 ..	129 ..	24
300 ..	100 ..	129 ..	23
400 ..	93 ..	—	20

F. C. Th.

**Admiralty gun-metal; Influence of increasing proportions of antimony and arsenic respectively upon the properties of —.** R. T. Rolfe. *J. Inst. Metals*, 1920, 24, 233—283.

IN sand-cast gun-metal the effect of a progressive increase in the antimony content is to produce a lowering of the strength and ductility, the alloy become harder and more brittle. With 1% Sb the maximum stress is about 15 tons per sq. in. and the elongation just under 7%. For material required to attain the Admiralty standard it is considered that 0.75% Sb is the maximum permissible. Up to 1.5% Sb does not appear to exert any definite influence on the soundness of the castings or on the microstructure. In chill castings the effects are similar. In the case of metal which has been twice melted, chill castings containing increasing amounts of antimony are increasingly unsound, especially with 1% or more of that element, and 0.75% is again considered to be the maximum figure allowable. The effect of arsenic on sand-cast gun-metal once melted is also to produce a progressive falling off of strength and ductility. For the maintenance of a safe margin over Admiralty specifications a maximum of 0.3% As should not be exceeded. Where the alloy is remelted the bad effects of the arsenic are not so pronounced, and the metal may be melted once and cast into ingots and a second time into castings. Up to 1.0% As does not appear to affect the soundness of gun-metal chill castings if these are melted once only. In the case of remelted metal chill castings become less and less sound as the arsenic content is raised, and a limit of 0.4% is suggested.—F. C. Th.

**Manganese bronze; Note on a failure of —.** J. H. Dickenson. *J. Inst. Metals*, 1920, 24, 315—332.

SOME peripheral projections of the end plate of a turbo-alternator rotor made of cast manganese bronze failed near soldered joints. The machine had run hot in service and the fracture of the bronze was clearly intercrystalline. Any fusible metal will cause manganese bronze to fail in a similar way provided that the fusible metal has been molten and that the bronze is under even a slight tensile stress. However tough the bronze is away from the solder, it will under these conditions act in its vicinity as a typical brittle substance. The

fusible metal passes between the crystals, which part without appreciable deformation. Although the bronze which failed was of the  $\beta$  type,  $\alpha$ -brasses will fail in a similar manner, and these alloys should not be used for the purpose mentioned or in electrical or other work where heating may occur and where solder or other fusible metal is present.

—F. C. Th.

**Tin; Precipitation of — by iron.** N. Bouman. *Rec. Trav. Chim.*, 1920, 39, 711—714. (*Of. J.*, 1920, 569 A.)

A REPLY to Kolthoff (*J.*, 1920, 747 A) in which the author gives an account of certain new experiments in support of his views.—W. G.

**Tin-antimony-copper alloys; Constitution and structure of —.** O. F. Hudson and J. H. Darley. *J. Inst. Metals*, 1920, 24, 361—371.

THE size of the cuboid  $\gamma$ -crystals depends on the rate of cooling and only indirectly on the casting temperature. The latter factor, however, determines the grouping of the primary  $\alpha$  constituent, whilst the size of these crystals is a function of the rate of cooling. It is possible that the effect of the casting temperature may be the result of some molecular arrangement in the liquid state, though it is more probable that convection and other currents by breaking up the grouping formed first play a very large part. Vigorous stirring during casting produces a structure like that which results from a low casting temperature even when the latter is high. The casting temperature should not exceed 50° C. above the temperature at which solidification commences, or, say, 300°—350° C. for an alloy containing 89% Sn, 8.7% Sb, and 2.3% Cu.

—F. C. Th.

**Crystal growth and recrystallisation in metals.** H. C. H. Carpenter and C. F. Elam. *J. Inst. Metals*, 1920, 24, 83—154.

THE work relates to aluminium and an alloy of tin and antimony containing about 1.5% Sb. This alloy is of special value for the study of crystal growth and recrystallisation, since if re-arrangement of the crystal system occurs on heating a polished and etched specimen to 150°—200° C. both the new and the old boundaries can be seen. The more important conclusions arrived at are: Neither crystal growth nor recrystallisation takes place unless the metal has been subjected to plastic deformation, i.e., they do not occur in castings. Further, they do not occur unless the metal is heated to a certain minimum temperature and for a certain time. The effects of work vary according to the degree of deformation, i.e., twin crystals or slip bands may be produced, but no crystal growth, or twins or slip bands may be produced with crystal growth on heating, or twins or slip bands may be produced, or entire recrystallisation on heating. The degree of deformation required to produce each of these types of effects is less as the temperature of heating is higher. There is no evidence that when equilibrium is reached the whole piece of metal will consist of a single crystal, but a certain definite degree of growth, dependent on the deformation, results for each temperature and time of reheating. Recrystallisation commences from a few points at the crystal boundaries of the old distorted crystals, and spreads through the mass. The recrystallisation is then followed by growth of the new crystals into each other, and crystals can both grow and be grown into at one and the same time. A large crystal may grow into a small one or vice versa. The orientation of the crystal does not affect crystal growth. Growth always occurs by slow boundary migration and not by sudden coalescence.

—F. C. Th.

*Metals; Crystallisation of — by electrolytic deposition and certain connected phenomena.* A. H. W. Aten and L. M. Boerlage. Rec. Trav. Chim., 1920, 39, 720—735.

THE conditions governing the form of the deposit obtained by electrolytic deposition of a metal from a solution of its salt are examined particularly for the case of the deposition of silver from a solution of its nitrate. The formation of different types of crystalline deposits or of a homogeneous layer is regulated by three factors, namely, number of nuclei, velocity of their growth, and variation of the polarisation tension with current density. The number of crystals per sq. mm. of cathode surface is practically a linear function of the intensity of the current, except when the current density becomes too great, the number being the greater the more dilute is the silver nitrate solution. It is only in the case where the polarisation tension is small that isolated crystals can be formed. The formation of a uniform deposit of a metal on an irregular object is regulated by the magnitude of the variation of the polarisation tension with the current density, a high value of  $\Delta e/\Delta i$  giving a homogeneous, flexible deposit. The addition of potassium nitrate, nitric acid, or gelatin to the solution of silver nitrate very considerably increases the number of crystals formed on a given area of the cathode.—W. G.

*Corrosive action of chlorine-treated water.* Clark and Isely. See XIXa.

#### PATENTS.

*Iron; Process for building up or growing of — upon steel or other surface.* S. O. Cowper-Coles. E.P. 154,232, 6.8.19 and 13.1.20.

THE required thickness of iron is deposited electrolytically from a hot saturated ferrous sulphate solution by immersing the part or parts to be restored in an electrolytic vat, the part which is not to be coated being protected by rubber or wax, or having the surface sulphided or oxidised so that any deposit is non-adherent. The deposited iron is annealed to produce soft tough iron which can be converted into steel by cementation. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 11,393 of 1889, 15,524 of 1900, 28,746 of 1910, 16,565 of 1913, 8795 of 1914, and 118,509; J., 1890, 812; 1911, 752; 1914, 650; 1918, 473 A.)—J. W. D.

*Ferrous metals; Electrical treatment of — during casting.* J. H. Wickett. E.P. 154,711, 3.9.19.

THE cavities in the moulding box are arranged in a more or less regular annulus, and a transformer core is inserted through the annulus out of contact with the molten metal and at an angle to the plane of the annulus, the core carrying a primary winding and the molten metal forming the secondary and being heated by the current induced in it.

—J. W. D.

*Puddled iron; Manufacture of —.* J. E. Fletcher. E.P. 155,042, 18.9.19.

PIG-IRON or pig-iron and iron or steel scrap melted in a furnace, or cold, is treated and partially refined in an open hearth refining furnace, and is subsequently finished and halled in puddling furnaces and the puddled iron is hammered or pressed or rolled by steam-driven power machinery. The open-hearth and puddling furnaces are heated by means of pulverised coal or a mixture of pulverised coal and oil, and the waste gases from the furnaces are utilised in heating the steam generators for the machinery.—J. W. D.

*Steel; Manufacture of —.* A. H. Henderson. U.S.P. 1,361,333, 7.12.20. Appl., 2.10.18.

METAL heated in a furnace or converter is brought

into contact with chloride of lime, anhydrous aluminium oxide, and anhydrous borax.

—J. W. D.

*Cast-iron; Transforming molten iron into pure — in the electric furnace.* Gräflich von Landsbergsche Elektrostahl und Metallwerke G.m.b.H. G.P. 309,252, 16.12.17.

MOLTEN iron that has been purified in the electric furnace is tapped into a ladle from which, after settling and holding back the slag, it is poured back into the furnace while at the same time a stream of finely divided charcoal or the like is charged in to carburise the metal.—A. R. P.

*Ores and oxygen compounds utilised as ores; Reduction of —.* F. M. Wiberg. E.P. 130,334, 23.7.19. Conv., 23.7.18.

THE ore, in particular iron ore, is reduced by a gas containing carbon monoxide which moves through the furnace in a direction opposite to that of the ore. The reduction process is divided into two stages, a portion of the gas being removed before passing through the whole mass of ore contained in the furnace and forced through a gas producer containing heated solid carbonaceous matter in order to regenerate carbon monoxide before re-introducing the gas into the furnace. The remainder of the gas passes through the whole charge of ore and exerts a further reducing action. The process may be carried out in a shaft furnace divided into three sections. A portion of the reducing gas is withdrawn at the upper end of the lowest section and is passed through a gas producer and then re-introduced at the bottom of the shaft. In the uppermost section the ore is heated to the reducing temperature by the combustion of gases rising from the lower sections of the shaft.

—J. W. D.

*Chromium or alloys of chromium; Production of —.* Aktiebolaget Ferroleggeringar. E.P. 135,187, 10.11.19. Conv., 15.11.18.

SILICO-CHROMIUM alloy containing more than 10% Si is bessemerised in a converter lined with substances containing chromic oxide, without the application of extraneous heat, in the presence of substances capable of forming silicates, whereby chromium alloys low in carbon and silicon are produced.

—J. W. D.

*Electric furnaces for the production of zinc.* B. Raeder, and A./S. Zink. E.P. 139,160, 3.2.20. Conv., 15.2.19.

THE furnace comprises three superposed chambers through which the charge passes successively. The electrodes, which are enclosed in tubes for protection from the flame in the flame or heating chamber, project from the top of the furnace through the upper drying chamber and intermediate heating chamber into the lower reduction chamber. The opening for the electrode in the roof of the reduction chamber serves also as an opening for feeding the charge, and the space round the electrode in the flame (heating) chamber is filled with pulverised charge to render the electrode opening gas-tight.

—J. W. D.

*Zinc from ores; Process of extracting —.* F. Laist and J. O. Elton, Assrs. to Anaconda Copper Mining Co. U.S.P. 1,362,166, 14.12.20. Appl., 5.1.20.

THE process is cyclical and consists of leaching the ore with a quantity of dilute sulphuric acid which is insufficient for complete extraction of the zinc, purifying the zinc sulphate solution, and producing from it by electrolysis metallic zinc and a dilute acid solution, a portion of which is returned to the ore-leaching operation, and the other portion

applied to leaching the residue from the primary leaching operation.—T. H. Bu.

*Furnaces; Oil fired [recuperative]* — J. Lambot. E.P. 141,055, 31.3.20. Conv., 3.4.19.

A FURNACE for melting metals is provided with detachable heads at each end, between the recuperator and melting hearth, the joints being made gas-tight by means of sand. The detachable heads carry detachable hollow plugs, each containing an adjustable oil burner mounted on an interior support, so that the oil flame can be directed downwards on the bath of metal.—B. M. V.

*Open-hearth furnaces; Reversible* — C. D. Fuller. U.S.P. 1,362,412, 14.12.20. Appl., 8.7.20.

IN a reversible open-hearth furnace means are provided for closing the side passages to the stack, and for producing a positive auxiliary circulation while the passages are closed.—W. J. W.

*Furnace; Soft-metal* — J. B. McClain. U.S.P. 1,362,429, 14.12.20. Appl., 16.8.20.

A FURNACE suitable for melting aluminium or other soft metals is provided with a bowl projecting downwards into the furnace body and spaced therefrom. The products of combustion rising from the firebox pass through an opening in the wall of the bowl and over the metal in it.—T. H. Bu.

*Furnace; Metal-working* — J. H. D. Hibbard. U.S.P. 1,362,532, 14.12.10. Appl., 1.11.19.

A METAL-WORKING furnace comprises a vessel with a number of pockets or containers. When the vessel is rocked the molten charge is poured from one pocket to another over the intervening dam, whereby reaction between the elements of the charge is promoted. When this reaction is completed the charge is discharged in solid form through a door.—T. H. Bu.

*Electro-deposited metals*. C. P. Madsen. E.P. 142,432, 5.8.19. Conv., 26.4.19.

"NICKEL metal" or substantially pure nickel is produced in an electro-deposition bath containing a sulphate and a chloride of the metal, an agent, e.g., sodium sulphate, adapted during the operation of electro-deposition to yield finely divided hydroxide of the metal, boric acid in solution, and a finely divided hydroxide in suspension. The temperature of the operation is 130° F. (54° C.) and the cathode employed is adapted to be intermittently or periodically exposed to a gaseous medium such as air, the time of exposure being not long enough to cause the successive deposits to separate.—J. W. D.

*Metals; Extracting — from metallic compounds, ores, foundry products, furnace dust, and the like*. F. Krupp A.-G. Grusonwerke. E.P. 144,728, 11.6.20. Conv., 3.6.19.

THE material to be treated is reduced to a fine state of subdivision and intimately mixed either dry or wet with sodium chloride also reduced to a fine state of subdivision, and the mixture heated in a furnace to the volatilisation temperature of the sodium chloride. The volatilised metal is collected. When the process is applied to lead zinc ores, lead and associated silver are volatilised, and the zinc remains in the residue.—T. H. Bu.

*Cleaning and coating metal articles*. N. Meurer. E.P. 154,235, 8.1.18.

THE articles are charged into a heat-insulated rotary drum, provided with apertures for removing the dust, and with inner projections adapted to lift the pieces under treatment and drop them through the drum space and then through the jet of coating metal which is introduced obliquely into the drum

from one of the ends. A shaft arranged crosswise to the drum in front of the injection opening carries the metal-squirting apparatus and also a sand jet blowing apparatus, these two devices being interchangeable.—J. W. D.

*Metals; Method of coating — [with aluminium]*. British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 154,808, 31.12.19.

THE metal to be coated, e.g., iron or copper, is pickled in acid, coated with tin in the presence of a flux, dipped in molten aluminium, then fired at about 800° C. in order to produce an alloy of aluminium with the original metal.—J. W. D.

*Copper; Production of coatings of — on iron*. Chem. Fabr. vorm. Weiler-ter Meer. G.P. 324,472, 26.6.17.

A SMALL quantity of gelatin, glue, or the like, is added to the copper-plating bath for the purpose of obtaining fine-grain deposits suitable for wire drawing.—L. A. C.

*Detinning of tinned iron scrap*. M. A. Adam, J. Stevenson, and A. T. Mahbitt. E.P. 154,242, 26.2.19.

THE tin is dissolved from the scrap by a strong solution of the chlorides of tin and iron, with or without a small percentage of hydrochloric acid, and the resulting solution is electrolysed, the speed of rotation of the cathode, the liquid flow, and the current density being so regulated that only a comparatively small amount of the total tin content is deposited from the solution and that the tin is deposited in non-coherent form. The solution is then used again for detinning.—J. W. D.

*Detinning*. W. C. Zacharias, Assr. to Steel Works Appliances Co. U.S.P. 1,362,381, 14.12.20. Appl., 16.8.19.

TIN-COATED articles are heated to a temperature at which the coating material becomes friable, and subjected while heated to such mechanical treatment as will effect disintegration and consequent detachment of the tin.—J. W. D.

*Copper; Production of spring or tempered* — S. O. Cowper-Coles. E.P. 154,373, 11.9 and 29.11.19.

COPPER is deposited electrolytically from a copper sulphate solution, to which an organic substance (e.g., glue or gelatin) or arsenic is added from time to time, on to a cathode or mandrel revolving at a critical speed of not less than 1000 peripheral ft. per minute. The greater the speed the harder is the deposited copper. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 21,974 and 26,724 of 1898, 26,211 of 1906, 30,377 of 1909, 23,775 of 1912, and 104,678; J., 1899, 1132; 1907, 1286; 1911, 137; 1917, 1277.)—J. W. D.

*Copper; Process of extracting — from its ores*. L. D. Mills, Assr. to The Merrill Co. U.S.P. 1,360,666, 30.11.20. Appl., 23.1.17.

IN a process of extracting copper, finely-divided freshly-reduced iron is employed to precipitate copper from its solution—A. de W.

*Sulphide ores containing lead and zinc; Treatment of —*. P. A. Mackay. E.P. 154,718, 4.9.19.

FINELY-GROUND sulphide ore containing lead is subjected to the action of sulphuric acid containing free sulphur trioxide in solution, or to the action of sulphur trioxide gas or vapour, whereby the lead is converted into lead sulphate. No external heat is supplied, the action being exothermic. The lead sulphate is subsequently separated from the un-



altered zinc sulphide by known methods. Bismuth, cadmium, silver, or the like when present are also converted into sulphates.—J. W. D.

*Mineral residues in a pulverulent condition; Conglomeration of —.* L. A. E. Irazusta. E.P. 155,522, 29.6.20.

Pure alumina and water are employed as binding agent in proportions varying according to the character of the mineral to be smelted, and the mixture is then formed into briquettes.—T. H. Bu.

*Ore-separator; Magnetic —.* H. W. Arnold. U.S.P. 1,362,072, 14.12.20. Appl., 17.11.17.

A MAGNETIC drum carrying a number of magnets enclosed in a water-tight casing revolves in a trough with an adjustable inlet apron for introducing water and material. An outlet apron with an adjustable scraper is arranged at the opposite side of the trough, and tailings are removed from the bottom by means of an adjustable gate with an adjacent partition for separating the tailings from particles on which the magnetic attraction is only slight.—T. H. Bu.

*Rust-proofing process.* E. P. Andrews. U.S.P. 1,362,213, 14.12.20. Appl., 22.12.19.

THE articles to be treated are subjected to an oxidising atmosphere at about 650° C. to form a coating of magnetic oxide, which is fixed by dipping the articles in a liquid medium.—J. W. D.

*Ores; Process of treating — and apparatus therefor.* C. S. Fogh. U.S.P. 1,362,408, 14.12.20. Appl., 17.7.19.

IN the treatment of ore with a gaseous reagent, a moving body of ore is preheated, subdivided, and the portions are delivered at various points along the line of movement.—W. J. W.

*Ore-reducing process and furnace.* E. H. Hamilton. U.S.P. 1,362,570, 14.12.20. Appl., 2.7.19.

A BLAST of air and finely divided fuel is introduced into the charge at a point within the interior wall of the furnace. By this means escape of the fuel up the walls of the furnace is avoided.—T. H. Bu.

*Ferro-tungsten; Process of treating — for the elimination of impurities.* P. M. McKenna. E.P. 146,108, 18.6.20. Conv., 11.8.16.

SEE U.S.P. 1,208,596 of 1916; J., 1917, 142.

*Manganese-steel articles; Method of and apparatus for hardening —.* F. Schaffer, Assr. to Chemical Foundation, Inc. U.S.P. 1,362,147, 14.12.20. Appl., 23.1.17.

SEE E.P. 112,986 of 1917; J., 1918, 154 A.

*Alloy steel; Manufacture of —.* R. A. Hadfield. U.S.P. 1,362,788, 21.12.20. Appl., 24.5.20.

SEE E.P. 131,980 of 1918; J., 1919, 826 A.

*Alloys of tungsten with other metals; Formation of —.* A. E. Alexander. From Metal and Thermit Corp. E.P. 155,739, 6.4.20.

SEE U.S.P. 1,350,709 of 1920; J., 1920, 694 A.

*Copper plates for process printing; Manufacture of —.* S. O. Cowper-Colcs. U.S.P. 1,362,082, 14.12.20. Appl., 15.3.20.

SEE E.P. 151,027 of 1919; J., 1920, 725 A.

*Combustion apparatus [for reduction of ores etc.]* R. J. Anderson, Assr. to International Fuel Conservation Co. U.S.P. 1,362,212, 14.12.20. Appl., 6.3.19.

SEE E.P. 150,761 of 1919; J., 1920, 714 A.

*Zinc solutions; Treatment of —.* F. Petersson, E. F. Petersson, administrator, Assr. to Metals Extraction Corp. U.S.P. 1,363,052, 21.12.20. Appl., 13.7.20.

SEE E.P. 152,752 of 1919; J., 1920, 823 A.

*Frath-flotation process; Apparatus for —.* L. A. Wood, and Minerals Separation, Ltd. E.P. 155,349, 11.9.19.

*Blast-furnace slag.* E.P. 139,173. See VII.

## XI.—ELECTRO-CHEMISTRY.

*Electrolytic cells.* Moore. See VII.

*Sodium permanganate.* Henke and Brown. See VII.

### PATENTS.

*Electric induction furnaces [; Three phase —].* Comp. Franç. des Métaux. E.P. 138,604, 20.1.20. Conv., 7.2.19.

AN induction furnace operating with three-phase current at 50 periods, and suitable for the fusion of metals and alloys, comprises three laminated cores each surrounded by a primary circuit. The primary circuits are surrounded by channels completely filled with metal, forming induction circuits and communicating with one another. Two of the channels are of different length from the third, which may be operated simultaneously with the others, or otherwise. The power factor is improved by flattening two of the channels in the form of horns. A cylindrical charging device closes from the air a chamber to receive the metal and communicating with the channels. A separate channel contains a resistor adapted to be heated by electric current.

—J. S. G. T.

*Electric furnaces.* L. Tagliaferri. E.P. 155,476, 2.2.20.

IN electric furnaces employing two- or three-phase current, in which the electrodes enter from both the top and sides of the furnace, the neutral wire is connected with a metal casing enclosing the furnace, whence the current is conveyed to one half of the electrodes by connecting leads.—J. S. G. T.

*Electrical etching; Composition for electrolytes for —.* J. H. Weeks, Assr. to J. S. and R. M. Weeks. U.S.P. 1,362,159, 14.12.20. Appl., 4.4.17.

AN anode carrying a resistant image to be etched is employed in conjunction with an electrolyte compound of iron sulphate and a mineral acid.

—J. S. G. T.

*Storage battery; Electric —.* Electrolyte for electric storage batteries. E. Hacking. U.S.P. (A) 1,362,294 and (B) 1,362,295, 14.12.20. Appl., (A) 17.12.19 and (B) 8.6.20.

(A) A SEPARATOR for electric storage batteries is formed by mixing an aqueous solution of sodium silicate and a solution of borax and sulphuric acid, with powdered pumice to the consistency of cream and allowing the mixture to solidify. (B) An electrolyte for storage batteries is composed of pumice and sulphuric acid.—J. S. G. T.

*Electrolytic cells.* I. H. Levin. E.P. 155,118, 31.12.19.

SEE U.S.P. 1,360,544 of 1920; J., 1921, 51 A.



**Electrolytic apparatus.** I. H. Levin. E.P. 155,457, 31.12.19.

See U.S.P. 1,360,542 of 1920; J., 1921, 51 A. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 12,765 of 1898, 21,557 of 1900, and 2124 of 1912.)

See also pages (A) 86, *Electric furnace* (E.P. 139,160), *Building up iron* (E.P. 154,282), *Casting ferrous metals* (E.P. 154,711); 87, *Copper* (E.P. 154,373); 91, *Waste micanite* (E.P. 155,318).

## XII.—FATS; OILS; WAXES.

**Chaulmoogra oil; Fractionation of —.** A. L. Dean and R. Wrenshall. J. Amer. Chem. Soc., 1920, 42, 2626—2645.

THE mixed fatty acids were distilled in 1-kg. portions from a 2-l. flask of the Claisen type, evacuated by a mechanical pump, and provided with a special receiver. The first 350 c.c. was worked up for hydrocarpic acid by fractional crystallisation from 80% alcohol and light petroleum, the next 300 c.c. was added to subsequent mixtures of crude acid, and the remaining distillate was treated for chaulmoogric acid by fractional crystallisation from 80% alcohol. The yields vary largely according to the quality of the oil, but even from low-grade oil 1 kg. of mixed acids yields at least 50 g. of pure hydrocarpic acid and 100 g. of pure chaulmoogric acid. (Cf. J.C.S., Feb.)—J. C. W.

**Cantaloup seed oil.** W. F. Baughman, D. Brauns, and G. S. Jamieson. J. Amer. Chem. Soc., 1920, 42, 2398—2401.

CANTALOUPE (*Cucumis melo*, L.) seeds were found to yield 30.4% of oil by extraction with ether. The cold-pressed oil had the following characters:—Sp. gr. at 25°/25° C., 0.9210;  $n_D^{25}$  = 1.4725; iodine value (Hanus), 125.9; saponif. value, 192.3; Reichert-Meissl value, 0.33; Polenske value, 0.26; acetyl value, 15.8; acid value, 0.43; unsaponifiable matter, 1.1%; soluble acids, 0.4% (as butyric acid); insoluble acids, 94.0% unsaturated acids, 80.2%; saturated acids, 14.3%. The oil consisted of glycerides of myristic acid 0.3%, of palmitic acid 10.2%, of stearic acid 4.5%, of oleic acid 27.2%, and of linolic acid 56.6%; unsaponifiable matter, 1.1%. No film developed even after 1 week when the oil was exposed to the air on a glass plate.—W. G.

**Catalytic activity of platinum [in hydrogenation]; Influence of lead on the —.** E. B. Maxted. Chem. Soc. Trans., 1920, 117, 1501—1506.

THE influence of the addition of successive small increments of lead, which was chosen as a typical non-volatile catalyst poison, on the subsequent activity of finely-divided platinum as a hydrogenating catalyst was studied by measuring the velocity of the absorption of hydrogen by a given weight of oleic acid under standard conditions in presence of a fixed amount of the catalyst and varying proportions of lead acetate dissolved in acetic acid. The depression of the catalytic activity of the platinum due to increasing proportions of lead was found to follow a linear law analogous to that observed for the inhibition of occlusion (J., 1920, 782 A). The view is developed that a certain proportion of the catalyst is poisoned by each milligram of lead added, the remaining catalyst functioning normally. This involves the assumption that the initial velocity of absorption induced by varying weights of catalyst under otherwise identical conditions, varies directly with the weight of platinum available for hydrogenation. This was shown experimentally to be the case.—G. F. M.

[*Cottonseed oil soapstock*;] *Application of super-centrifugal force [to recovery of oil from —].*

E. E. Ayres, jun. Chem. and Met. Eng., 1920, 23, 1025—1026. (Cf. Menzies, J., 1920, 231 r.)

By diluting soapstock, obtained as a by-product in cottonseed oil refining, until it is no longer gelatinous and then centrifuging it, a light emulsion and a heavy effluent are produced. The former contains oil globules of almost uniform size, 0.003 mm. diam., and closely packed together, whilst the latter comprises a few scattered globules, 0.002 mm. diam., By adding salt water to the light emulsion and re-centrifuging, a yield of about 60% of the oil in the soapstock may be recovered.—W. J. W.

**Mustard seed and substitutes.** I. Chinese colza (*Brassica campestris chinoleifera* Viehoveer). A. Viehoveer, J. F. Clevenger, and C. O. Ewing. J. Agric. Res., 1920, 20, 117—140.

A BOTANICAL and chemical study of seeds imported as rape seed, sold as mustard seed, and identified as Chinese colza, *Brassica campestris chinoleifera*, Viehoveer, n. var. The characteristics of the seeds have been established, and those which permit the identification and differentiation from true mustard seed are pointed out. The volatile oil from these seeds was identified as crotonyl isothiocyanate, an oil formerly found in rape, and not a suitable substitute for mustard oil in respect to condimental, bactericidal, or medicinal value. The yield varied from 0.4 to 0.6%, as compared with about 0.7—1.0% of volatile oil from true mustards, except *Sinapis alba*. The fixed or fatty oil expressed from the seed (yield 40—50%) showed the general characteristics of rape oil. It had sp. gr. 0.9097 at 25° C.; iodine value (Hanus), 100.3; saponif. value, 173.8; insoluble acids and unsaponifiable matter, 96.1%; soluble acids, 0.07%;  $n_D^{25}$  = 1.4695, solid acids, 19.52%; liquid acids, 75.52%. From the general composition of the seed and the character of the volatile oil it is suggested that the pressed oilcake may well be used as a stock feed. The plants, which are very vigorous and apparently hardy, offer possibilities as a forage crop.—W. G.

**Spermaceti and its examination for purity.** P. Bohrisch and F. Kürschner. Pharm. Zentralh., 1920, 61, 703—710, 719—726, 733—737.

STEARIC ACID, tallow, and paraffin, the usual adulterants of spermaceti, when present, alter the coarse leafy structure, rendering it small-leaved or granular, duller, and causing the spermaceti to lose its characteristic mother-of-pearl lustre. The sp. gr. of 21 samples of spermaceti obtained from wholesale and retail druggists in 1916 was determined; the values for trustworthy samples varied between 0.916 and 0.942, average 0.929. The determination of the sp. gr. does not, however, yield trustworthy results owing to the presence of air pockets in the sample as melted and cast for the determination. The limits of m.p. for the 21 samples examined were 44° C.—47.5° C. Spermaceti rapidly becomes rancid when exposed to light, but in metal or porcelain containers it may be kept for at least two years without becoming rancid. The so-called grease-spot test is of no value as a criterion of purity, since samples adulterated with 10% of stearic acid or tallow yield no grease spot when rubbed on paper. In the melted condition even the pure article greases paper. For the detection of paraffin in spermaceti the method of Branderhorst (Pharm.-Weekblad, 1909, 1043) is recommended. The sample (0.25 g.) is boiled with 5 c.c. of 10% potassium hydroxide solution in a test-tube, and 2—3 c.c. of cold distilled water is added. The presence of even 1% of paraffin is shown by the formation of an immediate clouding. Pure spermaceti also develops a cloudiness after one minute. For the detection of stearic acid Frerichs' modification of the ammonia test

(Apoth.-Zeit., 1916, 209) is best: 1 g. of spermaceti is warmed with 10 c.c. of ammonia in a test-tube until melted and is then thoroughly shaken. The cooled filtrate should show no separation on addition of dilute hydrochloric acid. The addition of acid may be omitted, as even in presence of 1% of stearic acid a cloudy filtrate is obtained. The acid and ester values of spermaceti are conveniently determined by a modification of von Hübl's method as follows: 3 g. of spermaceti is dissolved in 20 c.c. of light petroleum spirit in an Erlenmeyer flask, 5 c.c. of absolute alcohol added, and the mixture titrated with  $N/2$  alcoholic potash until permanently red to phenolphthalein. This gives the acid value. A further 25 c.c. of  $N/2$  alcoholic potash is added, the mixture carefully agitated, and allowed to stand in a stoppered vessel for 24 hrs. Titration with  $N/2$  hydrochloric acid gives the ester value. Genuine spermaceti shows an acid value up to 2.0, and a saponification value of 118–135. The iodine value serves as a measure of the sperm oil contained in a sample of spermaceti, commercial samples of the latter giving iodine values of 3.0–5.0. The preparation known as "spermaceti sugar," used as a remedy against coughs and hoarseness, is best prepared by moistening a mixture of spermaceti and sugar with alcohol and evaporating the solvent. Its purity is tested by extraction of the spermaceti with ether, and determination of the acid and saponification values on the sugar-free sample.

—A. de W.

*Soap solutions; Surface tension of certain —, and their emulsifying powers.* M. G. White and J. W. Marden. *J. Phys. Chem.*, 1920, 24, 617–629.

The cleansing power of soap is due to the fact that soap solutions have a low surface tension compared with that of pure water (but of approximately the same order as that of many oils) and not to the free alkali which is liberated by the soap when it is dissolved in water. Determinations of the surface tension of solutions of sodium stearate and sodium palmitate showed that this decreases with increasing concentration. The emulsifying power of solutions of sodium palmitate and sodium stearate for kerosene and linseed oil increases with increasing concentration of the soap solution, i.e., as the surface tension increases, the emulsifying power decreases. The presence of glycerin does not affect the surface tension to a considerable extent, and, therefore, the presence of glycerin should have no deleterious effect on the cleansing power of soap. The presence of large amounts of sodium carbonate in soap is undesirable, because this raises the surface tension and reduces the cleansing power.

—J. C. K.

*Sodium oleate solutions; Investigation of — in the three physical states of curd, gel, and sol.* M. E. Laing and J. W. McBain. *Chem. Soc. Trans.*, 1920, 117, 1506–1528.

A soap solution of one and the same concentration at any definite temperature may be prepared in the three characteristic states of clear fluid sol, transparent elastic gel, and white opaque curd. The sol and gel forms are identical in osmotic activity, concentration of sodium ions, conductivity, and refractive index, and differ only through the mechanical rigidity and elasticity of the gel. This proves that identical chemical equilibria and constituents are present in the two cases. The quantitative identity of conductivity in sol and gel is irreconcilable with all theories of gel structure hitherto advanced except the micellar theory of Nägeli. The formation of seep curd is analogous to a process of crystallisation, neutral soap separating from the solution in the form of curd fibres of microscopic or ultra-microscopic diameter. Coagulation and crystallisation are thus sharply distinguished from gelatin-

isation. The curd fibres consist of hydrated neutral soap, the hydration depending on the origin and previous history. Within corresponding limits their solubility is definite for each temperature, the so-called melting points being the temperatures at which the solubility curve rises to a value equal to that of the total concentration of the soap, and at which the last curd fibre just dissolves. The above results are of general applicability. A transparent commercial soap is a gel, all other hard soaps being gels containing a felt of curd fibres.—G. F. M.

#### PATENTS.

*Hydrogenation of unsaturated organic compounds [fatty acids and fatty acid esters]; Catalysts for use in —.* De Nordiske Fabriker De-no-fa Aktieselskab. E.P. 140,371, 13.1.20. Conv., 17.3.19.

CATALYSTS are prepared by precipitating aluminates of heavy metals, especially nickel aluminate, with a soluble aluminate, e.g., sodium aluminate, the heavy metal aluminate being then washed, dried, and reduced in a current of hydrogen at 300°–400° C. Alternatively nickel aluminate may be precipitated on an inert carrier, e.g., kieselguhr, and subsequently reduced.—A. de W.

*Oils and fats; Method of refining — whereby they are deodorised and their acidity is reduced.* K. H. Vakil. E.P. 155,020, 9.9.19.

A VEGETABLE, fish, or animal oil or fat is heated to 220°–300° C., and then brought into intimate contact with a current of carbon dioxide or a mixture of carbon dioxide and nitrogen, which may be at ordinary or higher temperature. For example, on heating coconut oil to 250° C. and passing carbon dioxide through it for 2 hrs., the acidity is reduced from 5.4% to 0.58% and the odour is completely removed.—A. de W.

*Soap and detergent; Process of making —.* M. Aisen, Assr. to Grain Soaps Corp. U.S.P. (A) 1,360,252, (B) 1,360,253, and (C) 1,360,254, 30.11.20. Appl., (A) 20.11.19, (B) 30.9.20, (C) 7.10.20.

(A) CEREALS are hydrolysed by subjecting them for 2 hrs. to a high temperature, below that of carbonisation (180° C.), anhydrous sodium carbonate or equivalent alkali carbonate in the proportion of 1 pt. to 2½ pts. of cereal is added, one-seventh by weight of caustic soda dissolved in water is sprayed into the mixture, and the mass is heated to the melting-point of the sodium carbonate crystals (below 100° C.) and maintained thereat for 1 hr. to effect saponification, after which any excess of sodium carbonate and caustic soda is neutralised with a fatty acid. (B) To the hydrolysed cereals from the heat treatment described above are added 1 pt. of a fatty acid to 1½ pts. of cereal, and caustic soda dissolved in water in the proportion of 1 pt. of caustic soda to 5 pts. of cereal. (C) A cereal flour is hydrolysed by means of a mineral acid and heat, the hydrolysed mass saponified by an alkali, and any free alkali then neutralised with a fatty acid.

—A. de W.

*Saponaceous cleansing composition; Method of manufacturing a —.* R. Gans. E.P. 132,511, 8.9.19. Conv., 24.6.18.

See G.P. 313,526 of 1918; J., 1920, 121A.

*Fatty acids from paraffin.* E.P. 142,507. See IIa.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Prussian blue; Decolorisation of — by oxidised oils.* F. Fritz. *Chem. Umschau*, 1920, 27, 242–243.

On cutting a section through a piece of blue or green linoleum, a banded structure can be fre-

quently observed, the face and back layers retaining their original colour, whilst the middle layer has changed to brown. On exposing the brown layer to air or, better, to air and light, the original blue colour is restored. The loss of colour is ascribed to the reduction of the Prussian blue to ferrous ferrocyanide,  $\text{Fe}_3\text{Fe}(\text{CN})_6$ , by the oxidised linseed oil. If a small quantity of Prussian blue be allowed to stand for a long time in contact with oxidised linseed oil in a test-tube, with frequent shaking, decolorisation of the pigment takes place if a sufficient amount of moisture is present. The examination of a section of blue or green linoleum can serve for the determination of the degree to which air has penetrated.—A. de W.

## PATENTS.

*Laquering; Process of* —. Soc. Anon. l'Helice Intégrale, anc. Etabl. L. Chauvière. E.P. 118,610, 23.7.18. Conv., 30.8.17

IN a process of laquering on an industrial scale in climates unfavourable to the rapid progress of hardening, the laquered articles are subjected in a dark chamber to the regulated action of heat, humidity, ozone, and electricity, the source of the latter two agents being disposed behind screens to protect the laquered articles from the ultra-violet rays. In this way hardening of the lacquer is effected in a reasonably short time.—A. de W.

*Japans [; Manufacture of black —].* British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 155,427, 5.11.19.

A NON-OLEAGINOUS protective colloid, e.g., a solution of glue, starch, agar, or water-glass, is added to the "water-japan" described in E.P. 121,533 (J., 1919, 81 A), and which consists of an emulsion in an ammoniacal solution of a japan base consisting of asphaltic material and a drying oil; e.g., to 5 galls. of japan base in 10 galls. of water containing 0.5 gall. of ammonia (sp. gr. 0.9) is added 10–20% by vol. of a 20% solution of glue. The colloid may be added before emulsification.—A. de W.

*Phenolic bodies; Condensation of — with aldehydic compounds.* Vickers, Ltd., The Ioco Rubber and Waterproofing Co., Ltd., and W. H. Nuttall. E.P. 154,656, 29.7 and 15.12.19.

A METALLIC carbide, nitride, silicide, phosphide, or cyanamide is used as condensing agent and to remove part of the water resulting from the reaction between a phenol and an aldehydic compound. For example, a mixture of 30 lb. of phenol, 27 lb. of paraformaldehyde, and 5 lb. of calcium carbide is heated to 100° C. until a resin of the desired consistency is obtained, further heating producing an insoluble and infusible product. Such condensing agents may also be employed only for the completion of the reaction, in which case the lower layer resulting from the reaction between a phenol and an aldehydic substance in the presence of a catalyst is separated and mixed with the condensing agent; e.g., 30 lb. of cresol, 30 lb. of 40% formalin, and 1 lb. of ammonium chloride are heated together in the usual manner, the lower layer separated, mixed with 4–8 oz. of powdered calcium carbide and warmed to 40°–50° C. until the heat of reaction is sufficient to complete the condensation.—A. de W.

*Varnish and other ingredients; Recovery of — from waste micanite and the like.* H. C. S. de Whalley, and The Micanite and Insulators Co., Ltd. E.P. 155,318, 5.7.19.

WASTE micanite, or other material in the form of sheets of insulating material united together by varnish under pressure, is heated in a dilute aqueous solution of ammonia or other alkali or salts of alkaline reaction, such as borax, by means of steam at 4–6 atm. pressure, whereby the varnish

is softened and the solvent penetrates between the surfaces of the sheets. Alternatively, the micanite may be treated with water in place of ammonia or other solvent, and the hot, wet material stirred into the cold alkaline solution. The resins are recovered from the alkaline solution after straining and, if desired after concentrating the same, by neutralising with sulphuric acid or the like, and heating the solution to facilitate the formation of a clot of melted resin which is then purified by remelting and washing.—A. de W.

*Painting, priming, and like compositions [emulsion paints]; Manufacture of —.* E. Schou, and Emulsion A./S. E.P. 155,398, 30.9.19.

AN "oleaginous material," e.g., 40 pts. of a drying oil, oil varnish, lacquer, resin or wax solution, etc., is emulsified in a suitable emulsifying agent, e.g., a thin aqueous solution or very fine mixture of 2 pts. of starch, casein, glue, etc. with 58 pts. of water, in homogenising apparatus of the type described in E.P. 140,147 (J., 1920, 377 A), or in other suitable apparatus, whereby the oleaginous particles are reduced to a diameter of 10 $\mu$  or less. Materials solid at ordinary temperatures are melted previous to emulsification. An increased "body" is obtained in the emulsified material by further addition of emulsifying ingredients, with or without the addition of suitable organic matter, or the emulsion may be concentrated by evaporation, osmosis, etc. of the water.—A. de W.

*Linseed oil substitutes [for paints]; Preparation of —.* D. Schoonderwaldt. E.P. 155,508, 26.5.20.

CRAZY American petroleum, e.g., fuel oil, 1 pt. by wt., is heated in an open cauldron and 1/25th pt. by wt. of methylated spirit, 1/10th pt. by wt. of lime from shells, and 1/10th pt. by wt. of fatty pipe-clay added. The mixture is boiled well for 2 hrs. with agitation, strained to remove coarse particles, and mixed with 1/10th pt. by wt. of stand oil.—A. de W.

*Resin; Process of producing —.* S. P. Miller, Assr. to The Barrett Co. U.S.P. 1,360,665, 30.11.20. Appl., 8.3.20.

THE polymerisable constituents in naphtha are polymerised with a polymerising agent below 20° C.—A. de W.

*Acid pots for making white lead.* E.P. 155,373. See VIII.

## XIV.—INDIA-RUBBER ; GUTTA-PERCHA.

*Caoutchouc; Action of sulphuric acid on natural and artificial varieties of —.* F. Kirchhof. Kolloid-Zeits., 1920, 27, 311–315.

COLD concentrated sulphuric acid converts raw rubber immediately into a hard, brittle mass which after several days becomes soft again. The action is accompanied by the evolution of sulphur dioxide, and the soft product becomes hard again when treated with water. Benzene solutions of raw rubber, gutta-percha, and artificial dimethyl-caoutchouc H, yield white solid substances which may be powdered and constitute oxidation products of the rubber. The action of sulphuric acid and the nature of the product are controlled by the nature of the solvent, by the quantity and particularly by the duration of the action of the acid, and by the concentration and nature of the rubber.—J. F. S.

*Rubber; Regeneration of — in its technical and economic relations.* P. Alexander. Chem. Ind., 1920, 43, 531–537, 543–545.

THE method of regeneration should be selected to suit the crude material and the requirements of

the finished "reclaimed" rubber. The operation is essentially one of heat treatment for the restoration of plasticity, but the removal of fabric from the rubber scrap is commonly necessary, and for this purpose the so-called "acid," "alkali," "solution" and "mechanical" processes are available; of these the "alkali" method is most widely adopted, but the "solution" process possesses an advantage in that not only are both fabric and rubber recovered, but a partial separation of the compounding ingredients from the latter is also possible. For the evaluation of reclaimed rubber it is important that chemical examination should be supplemented by physical tests. The value of aniline in the "reclaiming" process is probably merely as a softening agent for the rubber, and not at all as a desulphurising agent. Regenerated rubber has nothing to fear from the competition of synthetic rubber, and in view of the probable increase in the demand for rubber goods the "reclaimed rubber" industry will be of importance in the future.—D. F. T.

#### PATENTS.

*Rubber-proofed fabrics.* L. Minton. E.P. 155,469, 20.1.20.

*RUBBER-PROOFED* fabrics with a lustrous finish are produced by applying mica powder to the adhesive rubber surface before vulcanisation, or by incorporating mica in the rubber before this is spread on the fabric.—D. F. T.

*Rubber; Treatment of* —. Hunter Dry Kiln Co., Assecs. of H. Hunter. E.P. 138,915, 11.2.20. Conv., 25.9.15.

SEE U.S.P. 1,218,261 of 1917; J., 1917, 465.

*Rubber; Mills for grinding, crushing, or pulverising* —. S. Soksl. From Naaml. Vennoots. Versch. & Co.'s Scheepswrf en Machinefabriek. E.P. 132,789, 13.9.19.

*Gas-stopping linings.* E.P. 154,942. See V.

### XV.—LEATHER; BONE; HORN; GLUE.

*Bating; Mechanism of* —. J. A. Wilson. J. Ind. Eng. Chem., 1920, 12, 1087—1090.

THE mechanism of bating is shown to comprise reducing limed skins to a condition of minimum swelling, and digestion of the elastin fibres present in the outer layers of the skins.—W. P. S.

*Tannin of oak bark.* K. Feist and R. Schön. Arch. Pharm., 1920, 258, 317—318.

THIS tannin contains no methoxyl group, but is converted by diazomethane into a stable methyl derivative, which has  $[\alpha]_D^{20} = -43^\circ$  and molecular weight ca. 1800. A precursor of the tannin could not be discovered in the bark, but a preparation of catechin—which gives the same degradation products—was also found to be *lævo*-rotatory. (Cf. J.C.S., Feb.)—J. K.

#### PATENTS.

*Hides or skins; Curing* —. G. R. Olliphant. U.S.P. 1,362,502, 14.12.20. Appl., 6.4.20.

THE hides are soaked in an aqueous solution containing the leached constituents of "acid iron earth."—L. A. C.

*Refuse from tanneries and the like; Treatment of* —. H. C. Marris, and W. Walker and Sons, Ltd. E.P. 154,961, 1.8.19.

A MIXTURE containing ferric oxide or hydroxide prepared by treating spent vegetable matter (tan bark etc.) from tanneries or sawdust with a solution of

an iron salt, e.g., iron chloride or sulphate, and spent lime liquor, is suitable for absorbing sulphur compounds from coal gas or the like.—L. A. C.

### XVI.—SOILS; FERTILISERS.

*Soil air; Carbon dioxide of the* —. H. W. Turpin. Cornell Univ. Agric. Exp. Stat., Memoir 32, Apr., 1920, 319—362.

EXPERIMENTS with oat and millet plants showed that the plant itself, and soil organisms, produce most of the carbon dioxide in the soil; at the period of its most active growth the plant often produces many times as much carbon dioxide as is produced by the soil organisms. There is little relationship between temperature and carbon dioxide content of a soil bearing crops, but with bare soil the carbon dioxide content is usually high during warm weather. The excess carbon dioxide in a soil growing a crop is due to respiratory activity of the plants rather than to decay of root particles.

—W. P. S.

*Plants; Influence of the lime-magnesia ratio on the growth of* —. O. Loew. J. Landw., 1920, 68, 225—233.

ALTHOUGH there is some evidence that a lime-magnesia ratio of 1:1 or of 2:1, according to the nature of the soil, is the most favourable for the growth of plants (wheat and oats), pot cultures tend to give misleading results owing to crowding of the plants, interference with root growth, etc.

—W. P. S.

*Plants; Action of soda in the presence of potash as a food for* —. T. Pfeiffer, A. Rippel, and C. Pfotenhauser. J. Landw., 1920, 68, 255—284.

SODIUM salts may to some extent replace potassium salts as food for plants (cereals), and sodium is then found in the leaves and stalks.—W. P. S.

*Carbon dioxide; Assimilation of* — by green plants. P. Mazé. Comptes rend., 1920, 171, 1391—1393.

LEAVES were collected from a number of different species of trees and plants under the most varied atmospheric conditions and immediately distilled, without the addition of water, under reduced pressure at 60° C. In almost every case the distillate contained ethyl alcohol, acetaldehyde, and nitrous acid, but formaldehyde was never detected. In addition the leaves of maize and kidney beans, collected in very fine weather, gave acetylmethylcarbinol, elder leaves gave free hydrocyanic acid and glycollic aldehyde, and poplar leaves gave lactaldehyde and a substance which on oxidation yielded propionic acid. (Cf. J.C.S., Feb.)—W. G.

*Dicyanodiamide; Direct method for the determination of* — in [calcium] cyanamide and mixed fertilisers —. R. N. Harger. J. Ind. Eng. Chem., 1920, 12, 1107—1111.

DICYANODIAMIDE is precipitated completely when its aqueous solution is treated with silver picrate; cyanamide and urea do not yield a precipitate with the reagent, and do not interfere with the determination of the dicyanodiamide. To determine dicyanodiamide in commercial calcium cyanamide, 10 g. of the sample is shaken for 30 mins. with 300 c.c. of water, then rendered slightly acid with nitric acid (litmus indicator), 75 c.c. of 5% silver nitrate solution is added, the mixture diluted to 500 c.c., and filtered; 100 c.c. of the filtrate is treated with 15 c.c. of 5% silver nitrate solution and 100 c.c. of saturated picric acid solution, cooled in ice-water for 30 mins., the crystalline precipitate

collected on a weighed asbestos filter, washed with water saturated with the crystalline compound (e.g., a precipitate from a previous determination), afterwards with ether, dried at 100° C., and weighed. To the weight is added 0.0044 g. for each 100 c.c. of solution from which the crystals were filtered, and the result is divided by 5 to obtain the amount of dicyanodiamide present. In the case of mixed fertilisers, 20 g. is mixed with 50 c.c. of water, 100 c.c. of saturated barium nitrate solution is added, the mixture rendered alkaline with barium hydroxide solution, shaken for 30 mins., diluted to 500 c.c., and filtered; 100 c.c. of the filtrate is then treated as described. If the sample contains chloride, the mixture should be filtered after the addition of silver nitrate and before the picric acid solution is added.—W. P. S.

[Calcium] cyanamide; Changes taking place in — when mixed with fertilising materials. R. N. Harger. J. Ind. Eng. Chem., 1920, 12, 1111—1116.

When calcium cyanamide is mixed with fertilisers containing acid phosphate (superphosphate) and 5—10% of moisture, the cyanamide content decreases rapidly, dicyanodiamide being the chief product of the decomposition; calcium cyanamide is not, however, affected by dry superphosphate. Moisture alone is capable of decomposing calcium cyanamide slowly.—W. P. S.

Bacterial decomposition of cellulose. Groenewege. See V.

#### PATENTS.

Fertiliser, and process for making the same. P. C. Hoffmann, Assr. to Virginia-Carolina Chemical Co. U.S.P. (A) 1,360,401 and (S) 1,360,402, 30.11.20. Appl., 19.6 and 10.8.20.

A FERTILISER is prepared by treating calcium cyanamide with (A) concentrated or (B) dilute phosphoric acid.—W. J. W.

Peat; Treatment of — for making useful products. W. B. Bottomley. U.S.P. 1,355,732, 12.10.20. Appl., 19.5.19.

SEE E.P. 124,629 of 1918; J., 1919, 381 A.

### XVII.—SUGARS; STARCHES; GUMS.

Sugars; Changes in the polarising value of — during refining. A. F. Blake. J. Ind. Eng. Chem., 1920, 12, 1104—1107.

THE changes which occur in the relationship between polarisation, true sucrose, and invert sugar during the refining of raw sugar are due in part to the destruction of levulose by the action of heat and lime, and to the absorption of levulose in excess of dextrose by the bone-black.—W. P. S.

Glucose [dextrose]; Disodium phosphate as catalyst for quantitative oxidation of — with hydrogen peroxide. E. J. Witzemann. J. Biol. Chem., 1920, 45, 1—22.

THE oxidation of dextrose by hydrogen peroxide is accelerated by the presence of disodium phosphate. The product of oxidation is carbon dioxide, and the reaction is catalytic and quantitative.—J. C. D.

Inulin; Relationship of — to fructose [levulose]. Constitution of polysaccharides. I. J. C. Irvine and E. S. Steele. Chem. Soc. Trans., 1920, 117, 1474—1489.

INULIN yielded a dimethyl ether on treatment in sodium hydroxide solution with dimethyl sulphate. This was converted by further methylation with

methyl iodide and silver oxide into trimethylinulin, a colourless viscous syrup, soluble in organic solvents, which represents the limit of methylation of inulin, showing that the latter contains three hydroxyl groups in each  $C_6H_7O_5$  unit. On hydrolysis with 1% oxalic acid at 100° C. trimethylinulin is converted into a trimethylfructose belonging to the  $\gamma$ -series. For purposes of identification this was converted by way of its methylfructoside into tetramethyl- $\gamma$ -fructose which proved to be identical with the tetramethylfructose obtained previously by the hydrolysis of octamethylsucrose. The structural relationship between sucrose and inulin is therefore a close one. From the uniformity of the products formed in the above reactions it appears that inulin is an aggregate of  $\gamma$ -fructose residues, each ketone molecule having lost two hydroxyl groups in the formation of the polysaccharide.—G. F. M.

Erythro-dextrin; Individuality of —. J. C. Blake. J. Amer. Chem. Soc., 1920, 42, 2673—2678.

DEXTRINS which give deep-red colours with excess of iodine-water, preceded by little or no blue colour, that is, specimens which are comparatively poor in amylo-dextrin, are the best as sources of erythro-dextrin. After precipitating amylo-dextrin from the solutions by means of dilute alcohol, the erythro-dextrin fraction is separated as a heavy liquid by adding more alcohol. This is treated with ammonium sulphate, when the erythro-dextrin separates in a high degree of purity and in a form which is easily desiccated. The chief impurity which is thus removed is amylocellulose. (Cf. J.C.S., Feb.)—J. C. W.

Conversion of cellulose into dextrose. Irvine and Soutar. See V.

Carbohydrates of the pecan. Friedemann. See XIXA.

#### PATENT.

Beet-molasses; Process of purifying —. W. D. Bonner. U.S.P. 1,362,078, 14.12.20. Appl., 12.10.17.

SUFFICIENT tartaric acid is added to the diluted molasses to precipitate the bulk of the potassium as acid potassium tartrate, and, after separation of the precipitate, the purification is completed by evaporation of the solution.—L. A. C.

### XVIII.—FERMENTATION INDUSTRIES.

Alcoholic fermentation; Chemically defined catalysts in —. C. Neuberg and M. Sandberg. Biochem. Zeits., 1920, 109, 291—329.

FERMENTABLE sugars, carbonyl acids of the carbohydrate series, aldehydic and ketonic plant bases, quinone and natural dyes, nitro and nitroso compounds, hydroxylamine derivatives, organic and mineral disulphides, polysulphides, thio and selenous acids, reducible metallic salts, and elements were investigated in regard to their effect on alcoholic fermentation.—S. S. Z.

Bacterial decomposition of cellulose. Groenewege. See V.

Alcohols. Macht. See XX.

#### PATENT.

Yeast and like substances; Air-pressure filtering apparatus for pressing —. F. W. Littleton. E.P. 155,738, 1.4.20.

## XIXA.—FOODS.

*Milk; Influence of the diet of the cow upon the nutritive and anti-scorbutic properties of cow's milk. Vitamin studies.* R. A. Dutcher, C. H. Eckles, C. D. Dahle, S. W. Mead, and O. G. Schaefer. *J. Biol. Chem.*, 1920, 45, 119—132.

THE vitamin content of cow's milk is dependent upon the vitamin content of the cow's ration. The anti-scorbutic value of summer milk was found to be superior to that of winter milk.—J. C. D.

*Milk; Relation of fodder to the anti-scorbutic potency and salt content of —.* A. F. Hess, L. J. Unger, and G. C. Supplee. *J. Biol. Chem.*, 1920, 45, 229—235.

THE anti-scorbutic value of the milk and the percentage of certain inorganic and organic constituents may be influenced very greatly by the diet of the cow.—J. C. D.

*Milk; Comparison of some methods for determining the fat content of skimmed —.* T. J. McInerney and H. C. Troy. *Cornell Univ. Agric. Exp. Stat.*, Bull. 401, Jan., 1920, 69—85.

THE Adams gravimetric method, the Röse-Gottlieb method, and the Mojonier modification of the same yielded concordant and trustworthy results, as did also the Babcock method when modified as follows: At least 25 c.c. of sulphuric acid should be used, the temperature of the testing machine should be at least 180° F. (82° C.), the centrifuge disc should have a diameter of 15 in. and a speed of 1800 revs. per min., and the test should be centrifuged for 10, 2, and 1 min. periods.—W. P. S.

*Butter; Lecithin content of — and its possible relationship to the fishy flavour.* G. C. Supplee. *Cornell Univ. Agric. Exp. Stat.*, Memoir 29, Sept., 1919, 101—151.

THERE is in normal butter a sufficient amount (0.04—0.07%) of lecithin to yield, on decomposition, small quantities of trimethylamine, and this substance is essential for the manifestation of a fishy odour.—W. P. S.

*Butter; Phosphorus in —.* J. T. Cusick. *Cornell Univ. Agric. Exp. Stat.*, Memoir 30, Apr., 1920, 159—187.

DURING churning, about 25% of the total phosphorus present in the cream is retained in the butter, the remainder passing into the butter-milk, washings, etc. Butters prepared by the author contained 0.025—0.041% of P<sub>2</sub>O<sub>5</sub>. In storage the soluble organic phosphorus compounds break down, yielding inorganic phosphorus compounds. This change is to some extent controlled by the treatment of the milk and cream before churning, but, under certain conditions, bacterial action is the chief cause of the change. Salt has a marked effect in promoting protein decomposition in butter during storage. The new alcohol-soluble protein found by Osborne and Wakeman (J., 1918, 220 A) in milk is also present in butter. Decomposition of lecithin with the formation of trimethylamine is the cause of fishy flavour in butter (cf. *supra*). When this fishy flavour develops there is always an appreciable loss of soluble organic phosphorus.—W. P. S.

*Globulin of the cohune nut, Attalea cohune.* C. O. Johns and C. E. F. Gersdorff. *J. Biol. Chem.*, 1920, 45, 57—67.

THIS globulin resembles that derived from the coconut (J., 1920, 169 A), and contains a relatively high percentage of arginine and histidine.—J. C. D.

*Carbohydrates of the pecan.* W. G. Friedemann. *J. Amer. Chem. Soc.*, 1920, 42, 2286—2288.

PECAN (*Carya olivaeformis*) kernels contained: Moisture 3.75%, ash 1.70%, crude protein 12.27%, crude fibre 1.71%, ether extract 69.76%, and nitrogen-free extractives 10.81%. The oil in the crushed kernels was completely removed by extraction with ether, and the carbohydrates were determined in the resulting meal. The results obtained, calculated as percentages on the moisture-free pecan kernel, were: Sucrose 1.18%, invert sugars 2.88%, araban 1.95%, methylpentosans 0.22%, cellulose (crude fibre) 1.76%, amyloid 0.59%, tannins 0.33%, hemicellulose etc. 4.09%. There was no indication of the presence of xylan, mannan, or starch.—W. G.

*Humins formed by the acid hydrolysis of proteins.*

VI. *Effect of acid hydrolysis upon tryptophan.* G. E. Holm and R. A. Gortner. *J. Amer. Chem. Soc.*, 1920, 42, 2378—2385.

WHEN tryptophan is boiled with 20% hydrochloric acid it is slowly altered, and by prolonged hydrolysis parts of the molecule are broken down. In the absence of aldehydes or other reactive substances, tryptophan contributes but a very small fraction of its nitrogen to the "acid-insoluble" humin, but after boiling with acid for 144 hrs. a much larger amount of the tryptophan appears in the "soluble humin." As a protein hydrolysis rarely requires more than 24 hrs. boiling, it is very improbable that the "total" humin in the products of hydrolysis is derived from tryptophan without the intervention of some other reactive compound. Tryptophan is relatively easily deaminised by boiling with 20% hydrochloric acid, and probably some of the ammonia in the normal products of protein hydrolysis is derived from tryptophan. After hydrolysis of this amino-acid the distribution of the nitrogen is such that errors may be introduced into both the "basic" nitrogen and the "non-basic" nitrogen fractions of a Van Slyke determination (cf. J., 1911, 1135).—W. G.

*Mustard seed and substitutes.* Viehoveer and others. See XII.

*Phosphatides.* Brauns and MacLaughlin. See XXIII.

## PATENTS.

*Coffee substitutes; Manufacture of — from cereals and the malt of cereals.* K. Lendrich. E.P. 131,304, 12.8.19. Conv., 28.4.17.

CEREALS or malted cereals are uniformly moistened with water, which may contain a small quantity of sodium or calcium chloride, and treated with steam at a continually increasing pressure and with agitation, until the starch, albumin, and cellulose have been rendered sufficiently and uniformly soluble. The product is then roasted.—J. H. J.

*Grain; Apparatus for determining the amount of moisture in —.* J. S. Remington and J. P. Bennett. E.P. 154,315, 22.8.19.

GRAIN is fed through a spout on to the upper end of a shoot placed at such an inclination that the grain just slides down it. A weighted spring attached to the lower end of the shoot permits of slight variations in its inclination, such variations being indicated by a pointer on a scale or being recorded on a revolving drum. The angle of repose of grain increases with increasing moisture content.—J. H. J.

*Fish and the like; Cooling and freezing of —.* J. J. Piqué, and The Imperial Trust for the Encouragement of Scientific and Industrial Research. E.P. 154,250, 28.5.19.

FISH are placed in wire baskets which are simul-

taneously rotated on their axes and circulated in the inner compartment of a tank of cooled brine. The baskets are each provided with an inclined baffle on opposite sides, so as to effect a shuffling of the fish as the basket rotates. The brine is cooled by evaporation surfaces placed underneath the inner compartment and is circulated by paddle wheels, passing through an immersed filter in the course of its circulation.—J. H. J.

*Fish and the like; Cooling and freezing of —.* W. B. Hardy, J. J. Pique, and The Imperial Trust for the Encouragement of Scientific and Industrial Research. E.P. 154,663, 22.8.19.

A TANK contains a horizontal cylindrical chamber, the top of which is in the form of a lid, through which fish or other articles are introduced. A central rotating shaft running through the chamber carries three blades reaching to the circumference. An opening covered with gauze runs along the side of the chamber for the introduction of the cooling liquid, and another opening is provided in the bottom for its exit. Valves are provided in the tank for adjusting the height of the cooling liquid. The lid of the chamber opens into a gutter into which the fish are floated for removal after being rotated in the chamber for a sufficient period to effect freezing.—J. H. J.

*Food product and process for making same.* R. De O. McDill. U.S.P. 1,361,079, 7.12.20. Appl., 2.3.20.

SUFFICIENT sugar to act as a sweetener and preservative is added to citrus fruit juice and the mixture is dehydrated below 175° F. (79° C.) to a heavy paste.—J. H. J.

*Food products; Method of manufacturing —.* Food product and method of manufacturing the same. R. S. Fleming, Assr. to Merrell-Soule Co. U.S.P. 1,361,238 and 1,361,239, 7.12.20. Appl., 2.10.19.

A MIXTURE of fruit juice and a solution of glucose and dextrin produced by the hydrolysis of starch is atomised into a current of dry air, whereby the water is completely vaporised and the solids collect in the form of a dry powder.—J. H. J.

### XIXb.—WATER PURIFICATION; SANITATION.

*Water; Purification and sterilisation of — by means of lime in connexion with the supply of drinking water in Netherlands E. Indies.* J. Smit. Med. Geneesk. Lab. Weltevreden, 1920, 127—161.

A DARK BROWN moorland water on the east coast of Sumatra was treated with 1 g. of lime in the form of milk of lime per litre of water; after standing for 4 hrs. the number of bacteria was found to have been reduced to 40 per c.c., and after 10 hrs. to 6 per p.c. The water was neutralised by passing in carbon dioxide and then filtered. The filtrate was colourless and tasteless. In another water 0.1 g. of lime per litre was required to effect precipitation, leaving 0.084 g. per litre in solution, but the water was not sterile after standing for 24 hrs., and on further standing the alkalinity decreased and the bacteria increased. This result was due to the high temperature of the tropics. On infecting water with a culture of the cholera organism and treating with lime, no organisms were found in suspension at the end of 24 hrs., but the organisms in the precipitate were not all killed. Experiments were made to determine whether alum could be replaced by lime in the rapid filtration method of water purification. A turbid river water was mixed

with 50—200 mg. of lime per litre and immediately filtered through ordinary laboratory filter paper under sterile conditions, giving a clear and alkaline filtrate. The first portion of this was rejected, and the subsequent portion collected separately and neutralised with carbon dioxide. The neutral filtrate was tested bacteriologically and found to be nearly sterile. The precipitate on the filter paper contained living bacteria. For more polluted waters quantities of lime up to 300 mg. per litre had to be used. Clear river water and distilled water were used also with the same results, so that the important factors were alkalinity and filtration. Pathogenic organisms added to water were removed in the same manner as ordinary organisms. The same results were obtained when the treated water was filtered through a layer of sand.—J. H. J.

*Feed water for boilers; Purification of —.* G. Paris. Chim. et Ind., 1920, 4, 722—730.

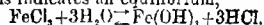
THE continuous purification method, using sodium carbonate, with continuous removal of the precipitated calcium carbonate, is recommended. The method effects considerable saving in heat, amount of sodium carbonate required, supervision, etc., compared with other methods. Diagrams are given showing the application of the method.

—W. P. S.

*Water; Corrosive action of chlorine-treated —.*

1. *Effects of steel on the equilibrium  $Cl_2 + H_2O \rightleftharpoons HCl + HClO$ , and of products of the equilibrium on steel.* G. L. Clark and R. B. Iseley. J. Ind. Eng. Chem., 1920, 12, 1116—1122.

IN the presence of chlorine, the most active agent in rusting iron in water is hypochlorous acid, which disappears first from the solution. Rust is formed rapidly during the disappearance of the hypochlorous acid and practically ceases to form when the concentration of hydrochloric acid becomes constant; this indicates an equilibrium,



The reaction  $H_2O + Cl_2 \rightleftharpoons HOCl + HCl$  proceeds very slowly and comes to equilibrium; iron has an apparent catalytic action on this reaction, resulting in the disappearance of both chlorine and hypochlorous acid, but as the iron is present almost completely as ferric ion its action must be almost entirely chemical and not catalytic, since the equilibrium is entirely destroyed.—W. P. S.

*Water analysis; Turbidity standard of —.* P. V. Wells. U.S. Bureau of Standards, Sci. Paper No. 367, 1920.

THE standard of turbidity adopted by the U.S. Geological Survey is shown to be inaccurate; this standard is a water containing 100 pts. per million of silica in such a state of fineness that a bright platinum wire of 1 mm. diam. can just be seen when 100 mm. below the surface of the water. The variations from the average may, in some cases, amount to 50%; this error could be eliminated largely by having all the standards prepared and distributed by some central authority.—W. P. S.

*Active carbonic acid in drinking water; Calculation and determination of —.* I. M. Kolthoff. Chem. Weekblad, 1920, 17, 390—396.

FROM a review of the available data, assuming dissolved calcium carbonate to be completely dissociated, and the hydrolysis of carbonate to bicarbonate only to influence the results, the values  $[OH^-] = 7.7 \times 10^{-6}$  and  $p_{OH} = 5.11$  at 16° C. were calculated, this result indicating that the dissolved carbonate is hydrolysed to the extent of 57%. Estimation of  $p_{OH}$  in a solution of pure carbonate confirmed these figures. On boiling, the bicarbonate formed by hydrolysis loses carbon dioxide, hydroxide being formed; after boiling for



$\frac{1}{2}$  hr., the hydroxyl ion concentration was found to have increased tenfold, the new value being  $7.7 \times 10^{-5}$ . A series of measurements of the solubility of powdered marble for various concentrations of carbon dioxide was carried out, carbon dioxide and bicarbonate being determined in the solutions after equilibrium was reached. The figures agreed fairly with the calculated, but were considerably lower than those determined from the table of Tillmans and Heublein. A new table is given, from which the active  $\text{CO}_2$  can be deduced, when  $\text{HCO}_3^-$ ,  $\text{CO}_3^{--}$ , and  $[\text{Ca}^{++}]$  are known. The values of Tillmans and Heublein hold only where  $[\text{Ca}^{++}]$  is equivalent to  $[\text{HCO}_3^-]$ , which is only true for drinking water which has no permanent hardness. (Cf. J.C.S., 1921, ii., 59).—S. I. L.

*Active carbonic acid in drinking water.* I. M. Koltzoff. Chem. Weekblad, 1920, 17, 558.

A REPLY to criticisms of the table put forward by the author (cf. *supra*). The values given in the table are stated to be not affected by the presence of such quantities of alkali or magnesium bicarbonates as would be found in drinking water, though if large quantities are present corrections must be made. The author's results are confirmed independently by Noll (J., 1920, 581 A), who also gives corrections for the presence of iron.—S. I. L.

*Sewage; Purification of — by the activated sludge process.* L. Cavel. Comptes rend., 1920, 171, 1406—1407.

THE author quotes experiments in support of his view that the purification of sewage by the activated sludge process is due to bacterial action. It is possible to shorten considerably the period of activation (normally 60 days). For example, by introducing some of the clinkers from a percolating bed, stopped seven months before, activation was accomplished in less than 15 days, and in one experiment in the laboratory the sludge was activated in 3 days.—W. G.

*Phenol; Chemical potential of — in solutions containing salts; and the toxicity of these solutions towards anthrax and staphylococcus.* J. S. Laird. J. Phys. Chem., 1920, 24, 664—672. (Cf. J., 1920, 799 A).

THE toxicity of a solution containing salt and phenol is the same as that of a solution containing phenol alone if the two solutions are in equilibrium with the same solution of phenol in kerosene. The use of this hydrocarbon offers the advantage over toluene that in the equilibrium experiments only a small proportion of the phenol passes into the upper layer. By the addition of salts the toxicity of a 4% phenol solution is altered owing to the change in the chemical potential of the phenol. —J. C. K.

*Germicidal value of some of the chlorine disinfectants.* F. W. Tilley. J. Agric. Res., 1920, 20, 85—110.

COMPARED on a basis of weight of chloramine T (sodium toluene sulphone chloramide; Dakin, J., 1916, 651) as against weight of chlorine as sodium hypochlorite (Dakin's solution; cf. J., 1915, 919) or hypochlorous acid (eusol; cf. Rettie, J., 1918, 23 T), or as chlorine in aqueous solution, chloramine T is less efficient than the others. If, however, the comparison is made on the basis of "available chlorine" it is much more efficient against *Staphylococcus aureus*, much less efficient against *B. pyocyaneus*, and approximately equal in efficiency against *B. typhosus*. These chlorine disinfectants are apparently of little value against *B. tuberculosis*. Experiments upon anthrax spores indicate that the germicidal action of chlorine compounds is not always so speedy as is commonly supposed, but

may extend over several days. The addition of ammonia to solutions of chlorine or hypochlorites very greatly increases their germicidal activity and tends to prevent depreciation in value on the addition of organic matter.—W. G.

*Poisoning by nitrohalogen derivatives of methanes.* A. Mayer, Plantefol, and F. Vlès. Comptes rend., 1920, 171, 1396—1397.

A DESCRIPTION of the symptoms produced in the human organism when substances like chloropicrin, bromopicrin, or dichlorodinitromethane are inhaled or injected intraperitoneally. (Cf. J.C.S., Feb.) —W. G.

*Air contaminated with poisonous gases; Use of liquid sprays for purifying —.* A. Desgrez, H. Guillemard, and A. Savès. Chim. et Ind., 1920, 4, 814—817.

A FULLER account of work, the chief results of which have been given previously (J., 1921, 59 A).

*Bacterial decomposition of cellulose.* Groenewege. See V.

#### \* PATENTS.

*Waste organic substances; Process of and apparatus for treating —.* A. MacLachlan, Assr. to F. G. Reigart. U.S.P. 1,360,427, 30.11.20. Appl., 16.4.18.

ORGANIC waste is placed in closed cylinders connected in series and is submitted to the action of an oxidising gas which is admitted to the first cylinder of the series below the level of the waste. The gas from the first cylinder is passed to the bottom of the second cylinder, and so on. Oily matter is precipitated upon the heavier oxidised waste.—J. H. J.

*Sewage and like liquids; Separating and removing semi-solids from —.* S. H. Adams. E.P. 154,370, 11.9.19.

THE crude sewage flows at a reduced speed over a perforated floor through which the sand and other heavy solids fall into a conical chamber below, where the solids pass directly into a displacement ejector worked by compressed air. The sewage passes on at an increased rate of flow to a second conical chamber into which it is directed in a downward direction, so that the flocculent matter and other solids fall to the bottom into an ejector, which is in the form of a continuous length of pipe, while the sewage flows upwards through a perforated screen in the upper portion of the chamber. Instead of using two chambers the whole operation may be conducted in one chamber, in which case the sewage passes downwards into the chamber and upwards through the perforated screen.—J. H. J.

*Water softening.* G. L. Borrowman. E.P. 155,092, 5.11.19.

SEE U.S.P. 1,348,977 of 1920; J., 1920, 671 A.

*Clarifying liquids.* E.P. 154,766. See I.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Cinchona series; Syntheses in the —.* VI. Amino-azo and hydroxyazo dyes derived from certain 5-amino cinchona alkaloids and their quinoline analogues. W. A. Jacobs and M. Heidelberger. J. Amer. Chem. Soc., 1920, 42, 2278—2286.

5-AMINOHYDROQUININE readily couples with diazotised aromatic amines to give crystalline azo dyes in which the amino group is remarkably labile. Most of these aminoazo dyes are red and give orange-red solutions in neutral solvents and deep purple solu-

tions in dilute acids. On boiling the solution in mineral acid the purple colour rapidly changes to brownish-red, the amino group being replaced by a hydroxyl group. Similar results were obtained with 5-aminodihydroquinidine and 5-aminoethylidihydrocypresine, amino-optochin. The lability of the amino group is apparently in part a function of the quinoline nucleus, as 5-aminoquinoline gives azo dyes which behave in a similar manner though somewhat less readily. The presence of a methoxy or ethoxy group in position 6 facilitates the replacement of the amino group.—W. G.

*Codeine; Two new reduction products of* —. C. Mannich and H. Löwenheim. Arch. Pharm., 1920, 258, 295–316.

ATTEMPTS were made so to modify the partially reduced benzene nucleus of morphine and codeine as to permit the normal application of the usual methods for determining constitution, since these give anomalous results when applied directly. Dehydroxydihydrocodeine,  $C_{17}H_{21}O_4N$ , m.p.  $107^\circ C$ ,  $[\alpha]_D^{25} = -81.47^\circ$ , obtained as the chief product of reduction of  $\beta$ -chlorocodide in presence of palladium, behaves normally when subjected to degradation through exhaustive methylation, and gives dehydroxydihydrocodomethine,  $C_{17}H_{23}O_4N$ , m.p.  $86^\circ C$ , which in turn similarly yields 3-methoxy-5-vinyltetrahydrophenanthrylene oxide. Dehydroxytetrahydrocodeine,  $C_{17}H_{23}ON_2$ , m.p.  $144^\circ$ – $145^\circ C$ ,  $[\alpha]_D^{25} = -36.92^\circ$ , is a secondary product of the above reduction and is also obtained by Clemmensen's method (J., 1913, 711; 1914, 217) from dihydrocodeinone,  $C_{17}H_{21}O_4N$ , m.p.  $193^\circ$ – $194^\circ C$ , which is itself prepared by reducing codeine in presence of palladium. (Cf. J.C.S., Feb.)—J. K.

*Angostura alkaloids.* J. Troeger and K. Bönicke. Arch. Pharm., 1920, 258, 250–277.

THE existence of galipidine and cusparidine could not be confirmed by Troeger and Kroseberg (J., 1912, 1050), and it is now shown that the former is an impure form of galipine, and further that the formula of this base is  $C_{17}H_{21}O_4N$ . Like cusparin (Troeger and Müller, J., 1915, 449), it suffers demethylation when heated in dry hydrogen chloride, giving a phenol,  $C_{17}H_{21}O_4N$ , m.p.  $225^\circ C$ , and it is isomerised to isogalipine when gradually heated in a stream of methyl iodide vapour from  $130^\circ$  to  $190^\circ$ – $200^\circ C$ . (Cf. J.C.S., Feb.)—J. K.

*Hyenanchin and other constituents of Hyenanche globosa.* T. A. Henry. Chem. Soc. Trans., 1920, 117, 1619–1625.

By extraction of the stems, leaves, and fruits of *Hyenanche globosa* (a South African plant of the order *Euphorbiaceae*) with hot 95% alcohol, after removal of wax etc. by percolation with hot chloroform, a thick syrup was obtained, from which two crystalline isomeric toxic substances, of the probable formula,  $C_{17}H_{21}O_4$ , were isolated, to which the names hycnanchin and isohyenanchin are given. Hyenanchin is probably a dilactone, and is converted by the regulated action of weak alkali into a dihydroxydicarboxylic acid,  $C_{17}H_{21}O_6$ , which is unstable and readily loses a carboxyl group. It has a physiological action almost identical in kind with that of picrotoxin, but much weaker, and probably belongs to the same group of convulsant non-nitrogenous poisons as picrotoxinin and tutin. A new wax alcohol,  $C_{17}H_{33}OH$ , m.p.  $82^\circ$ – $83^\circ C$  (corr.), and a new phytosterol,  $C_{27}H_{48}O$ , m.p.  $265^\circ C$  (corr.), were isolated from the chloroform extract of the leaves and stems, and a yellow colouring matter of the flavone group from the ethereal extract of the tannin.—G. F. M.

*Cocaine solutions; Influence of the reaction of — on their stability.* A. Rippel. Arch. Pharm., 1920, 258, 287–295.

THE activity of cocaine solutions of definite acidity or alkalinity after sterilisation by heat was determined by physiological tests. Boiling for one hour in neutral solution or in a solution of the same alkalinity as the blood caused diminutions in activity of 60–70% and 80% respectively, but no deterioration occurred in solutions varying in acidity from  $pH=1$  to  $pH=5.8$ . Similar, but less rapid, changes took place at the ordinary temperature. (Cf. J.C.S., Feb.)—J. K.

*Santonin; New source of* —. H. G. Greenish and C. E. Pearson. Pharm. J., 1921, 106, 2–3.

ABOUT 1% of santonin was found to be present in the leaves of *Artemisia brevifolia*, a plant growing abundantly in Cashmir and Western Tibet at an altitude of 9000–14,000 ft.—W. P. S.

*Anti-body studies. III. Chemical nature of bacterial anti-bodies.* F. M. Huntton, P. Masucci, and E. Hannum. J. Amer. Chem. Soc., 1920, 42, 2654–2661.

THE anti-bodies of pneumococcus serum have been isolated as far as possible and the solution tested with protein reagents. It appears that they do not belong to the class of serum proteins. (Cf. J.C.S., Feb.)—J. C. W.

*Arsphenomine (3,3'-diamino-4,4'-dihydroxyarsenobenzene dihydrochloride); Hypophosphorous acid preparation of* —. W. G. Christiansen. J. Amer. Chem. Soc., 1920, 42, 2402–2405.

THE reduction of 3-amino-4-hydroxyphenylarsinic acid gives a salvarsan (Fargher and Pyman, J., 1920, 465A) which is relatively non-toxic. This furnishes a means of converting toxic arsphenamine (salvarsan) into a relatively non-toxic form by oxidation and subsequent reduction with hypophosphorous acid. Although the product obtained by this method is less readily soluble in water than that obtained by the methyl alcohol-ether method, the use of warm water in dissolving it has not been found to be injurious. The source of the amino-hydroxyphenylarsinic acid does not affect the toxicity of the arsphenamine obtained from it.

—W. G.

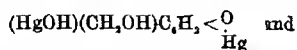
*Mercury derivatives of phtholeins.* E. C. White. J. Amer. Chem. Soc., 1920, 42, 2355–2366.

MERCURY derivatives may be prepared from the phthalcins either by the action of yellow mercuric oxide on a solution of a phthalein salt or by the action of mercuric acetate on the free phthalein in alcoholic solution or aqueous suspension. Where an alcoholic solution is used in the second method an acetoxy-mercury derivative,  $R.HgOOC.CH_3$ , is formed. The mercury apparently enters the phenolic group of the phthalein molecule in the ortho position to the hydroxyl group or the quinone oxygen. The number of atoms of mercury entering the molecule is limited by the number of such free ortho positions, and if all four are occupied, as in Eosin, no substitution of mercury takes place. Mercury derivatives containing from one to four atoms of the metal have been prepared from the following phthaleins: phenolphthalein, o-cresolphthalein, fluorescein, dibromofluorescein, and phenolsulphonphthalein. In the determination of mercury in these compounds, the organic matter is destroyed by potassium permanganate and sulphuric acid and the mercury subsequently precipitated as sulphide and weighed. These compounds appear to be of some value in the treatment, both internally and externally, of genito-urinary infec-

tions and of syphilis. Identity of composition may or may not mean identity of biological behaviour if the two samples have not been made by precisely the same procedure.—W. G.

*Mercury compounds of some phenylcarbinols.*  
M. C. Hart and A. D. Hirschfelder. J. Amer. Chem. Soc., 1920, 42, 2678—2686.

SALIGENIN and *m*-nitro-*p*-hydroxyphenylcarbinol react with boiling mercuric acetate solution to form very insoluble mercury compounds of the formulae



$(\text{HgOH})(\text{CH}_2\text{OH})\text{C}_6\text{H}_4(\text{NO}_2)\text{OH}$  respectively. These yield insoluble salts with acids, but soluble salts with alkali hydroxides. They are about as powerfully antiseptic as mercuric chloride, but are far less irritant, and they are being used successfully in the treatment of gonorrhoea. (Cf. J.C.S., Feb.)—J. C. W.

*Cyclohexane; Preparation of certain derivatives of*  
— A. E. Osterberg and E. C. Kendall. J. Amer. Chem. Soc., 1920, 42, 2616—2626.

THE preparation of cyclohexane derivatives on a large laboratory scale is described. Cyclohexane and cyclohexanol are obtained by reducing benzene and phenol by hydrogen under 115–120 atm. pressure in steel bombs at 250° C., the catalyst being nickel. Cyclohexanol is used as the source of cyclohexanone and cyclohexene. Cyclohexylamine is made from cyclohexanone-oxime, and 2-chlorocyclohexanol by the action of hypochlorous acid on cyclohexene. Chlorocyclohexanol is converted into aminocyclohexanol and this into 2-halogenocyclohexylamines. (Cf. J.C.S., Feb.)—J. C. W.

*Dichloroacetic acid; Preparation of — from chloral.* G. W. Pucher. J. Amer. Chem. Soc., 1920, 42, 2251—2259.

DICHLOROACETIC acid may be prepared on a large scale by adding 400 g. of commercial sodium cyanide dissolved in 1100 c.c. of water to a solution of 1 kg. of chloral hydrate in 3 l. of water at 40° C., with constant stirring. The addition should be at such a rate that the temperature does not rise above 50° C. After one hour the water is evaporated off, the crude sodium salt is suspended in benzene, and dry hydrogen chloride is passed into the ice-cold suspension. The precipitated sodium chloride is filtered off and the dichloroacetic acid is recovered from the filtrate after distilling off the benzene. A yield of 60–65% of the calculated amount is obtained. Various metals were examined as to their resistance to corrosion by this acid. Aluminium appears to be the most resistant to the pure acid at 90° C., whereas the dilute acid attacks it readily, but does not attack copper or lead appreciably.—W. G.

*Methylamines from methyl alcohol and ammonium chloride.* W. D. Turner and A. M. Howald. J. Amer. Chem. Soc., 1920, 42, 2663—2665.

A MIXTURE of ammonium chloride (1 mol.), zinc chloride (1.5), and methyl alcohol (12 mols.) heated at 303° C. for 8 hrs. in a steel tube gave yields of 55.0, 7.5, and 1.86% respectively of mono-, di-, and trimethylamines, calculated on the weight of ammonium chloride. A mere trace of amine is formed in the absence of a dehydrating agent.—J. C. W.

*Ethyl iodide; Preparation of* — B. E. Hunt. Chem. Soc. Trans., 1920, 117, 1592—1594.

THE preparation of ethyl iodide is more successfully accomplished by using 80% instead of absolute alcohol, and the use of a large excess is unnecessary. The modified procedure is to add during the course of about 20 mins. 50 g. of red phosphorus to a

mixture of 500 g. of iodine and 281 g. of 80% alcohol (by weight) in a 4 l. flask. The reaction mixture is then boiled gently for 2½ hrs. on a water bath, and the ethyl iodide thereupon distilled off, and purified in the usual way. The yield amounts to 92% of the theoretical. The main reaction appears to proceed according to Beilstein's interpretation with the formation of phosphoric acid, and not phosphorous acid as usually stated. The use of a few drops of thiosulphate solution in purifying ethyl iodide gives a product which darkens less rapidly than when sodium carbonate alone is used.—G. F. M.

*Ether; Catalysts in the manufacture of* — H. Schlatter. J. Ind. Eng. Chem., 1920, 12, 1101—1102.

THE addition of aluminium sulphate offers no advantage in the manufacture of ether and is actually harmful since it causes pitting and rapid failure of the coils; the slight catalytic effect of the aluminium salt is not greater than that produced by the lead sulphate normally present in lead ether stills.—W. P. S.

*Formaldehyde; Commercial* — G. F. Merson. Pharm. J., 1921, 106, 4.

A SUPPLY of commercial formaldehyde solution received from the United States contained over 13% of proof spirit (ethyl alcohol); the use of ethyl alcohol in place of the usual methyl alcohol as a "filler" was evidently made for economic reasons.—W. P. S.

*Alcohols; Toxicological study of* — D. I. Macht. J. Pharm. Exp. Ther., 1920, 16, 1—10.

IN the normal aliphatic series (methyl, ethyl, butyl, and amyl alcohols) the toxicity increases with the mol. wt. Secondary (propyl, butyl, and amyl) alcohols are less toxic than the corresponding primary alcohols.—J. C. D.

*Thymol; Determination of — in Spanish thyme oil.* H. Mastbaum. Chem.-Zeit., 1921, 45, 18—19.

SPANISH thyme oil is distilled from four varieties of thyme, but only one of the latter, *Orydthymus capitatus*, yields an oil containing a high percentage (67%) of total phenols; the oils obtained from common thymes, *T. vulgaris*, *T. Zygis*, and *T. hemalis*, contain about 35% of total phenols (thymol and carvacrol). The quantity of the latter may be determined by shaking the oil with 5% sodium hydroxide solution and measuring the volume of residual oil; the difference between this and the volume of oil taken gives the volume of the total phenols present. To determine thymol, 25 g. of the oil is shaken for 30 mins. with 100 c.c. of 5% sodium hydroxide solution, the alkaline solution is drawn off, and the extraction repeated with 80 c.c. and 50 c.c. of sodium hydroxide solution. The united alkaline extracts are diluted to 250 c.c., filtered, 200 c.c. of the filtrate is acidified with concentrated hydrochloric acid, and cooled in ice-water; the crystallised thymol is collected on a small suction filter, the oily portion (carvacrol) is drawn off as much as possible, and the crystalline mass pressed between filter paper and weighed.—W. P. S.

See also pages (A) 78, *Explosion limits of acetaldehyde* (Jorissen); 89, *Chaulmoogra oil* (Dean and Wrenshall), *Chinese colza seed* (Viehoever and others); 102, *Phosphatides* (Brauns and MacLanghin), *Methoxyl group* (Troeger and Tiebe).

#### PATENTS.

*Chlorinating hydrocarbons; Process for* — [Preparation of trichlorhydrin.] H. F. Saunders and L. T. Sutherland, Assrs. to The Glysyn Corp. U.S.P. 1,362,355, 14.12.20. Appl., 31.8.20.

TRICHLORHYDRIN is prepared by the action of

chlorine on propylene chloride in the presence of a gaseous compound of sulphur as catalyst.—D. F. T.

*Acetic aldehyde; Manufacture of*—H. Dreyfus. U.S.P. 1,361,974, 14.12.20. Appl., 4.6.17.

SEE E.P. 115,899 of 1917; J., 1918, 443 A.

*Oxygen compounds from paraffin*. E.P. 142,507. See IIa.

*Extracting ethylene*. E.P. 147,543. See IIa.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Photographic plate; Action of light on the*—G. I. Higson. Phot. J., 1921, 61, 35–44.

THE equations previously deduced by Slade and Higson (J., 1921, 27 A) are further examined in relation to the ordinary characteristic curve. General curves are deduced for the two equations for intensity scale exposures when  $I$  is large and when  $I$  is small, and also for the equation for time scale exposure when  $t$  is moderately large. These curves are similar to the characteristic curve, and a mathematical analysis of their properties leads to conclusions as to constancy of inertia, occurrence of under-exposure period, central speed method of plate testing, etc., which are in agreement with the results deduced directly from the characteristic curve. There is also a short examination of the problems which arise in passing from the case of a thin film of emulsion containing grains of uniform size and speed to ordinary emulsion films, several grains in thickness, the grains varying also both in size and speed.—B. V. S.

[*Photographic*] *characteristic curve; Interpretation of a — with the aid of its first derivative*. F. F. Renwick. Phot. J., 1921, 61, 10–12.

THE characteristic curve is obtained by plotting densities ( $D$ ) against logarithms of exposures ( $\log E$ ). If instead of density the rate of increase of density ( $dD/d \log E$ ) is plotted against  $\log E$ , thus getting the first derivative of the characteristic curve, an ordinate of the new curve is proportional at any point to the amount of silver bromide rendered developable by the exposure at that point, but which is not developable at a smaller exposure. It is suggested that the use of these curves and a study of their meaning will assist materially in the solution of various photographic problems.

—B. V. S.

*Soluble iodides; Action of — on photographic plates*. F. F. Renwick. Phot. J., 1921, 61, 12–15.

THE action on photographic plates of soluble iodides in sufficient quantity to convert all the silver bromide into iodide has been previously described (J., 1920, 156 r). Very weak solutions, however (1:50,000) produce a definite colour-sensitising which explains the supposed fogging action which had previously been ascribed to such solutions. Of other salts tested only cyanides, in strengths from 1:2000 to 1:10,000, behaved similarly. Spectrographs showing the added sensitiveness are given.

—B. V. S.

*Sensitiveness of silver bromide; Reduction of — by amidol and allied bodies*. Lüppo-Cramer. Phot. Ind., 1920, 505–506.

USING 1/2000 solutions, amidol reduces the sensitiveness to 1/200, triaminotoluene to 1/600, triaminobenzene and triaminophenol to 1/60–1/70, diaminoresorcinol less still, and metol to 1/3. Plates bathed with erythrosin still retain some colour-sensitiveness after treatment with amidol, so that the method of development previously recom-

mended, using a bright yellow light (J., 1921, 28 A), is not applicable to such plates.—B. V. S.

*Sensitiveness of silver bromide; Action of developing solutions on —*. Lüppo-Cramer. Phot. Ind., 1920, 664–668.

ALKALINE solutions of quinol, metol, and edinol free from sulphite desensitise considerably, though less than amidol (*cf. supra*), pyrogallol solutions desensitise much less, and *p*-aminophenol, glycin, and catechol hardly at all. Plain aqueous solutions behave similarly if allowed to oxidise. Sulphite, in as low concentration as 3.5%, almost completely prevents the desensitising action, but the effect is only temporary, desensitising starting as soon as the oxidation of the sulphite allows formation of developer oxidation products. The effect is therefore due to interaction between the silver salt and the oxidation product. Quantitative measurements were made with quinol (*i.e.*, with its oxidation product, quinone), and with metol. The fact that strongly oxidised amidol solution is less effective than a fresh, slightly oxidised solution is explained as due to the existence in the former case of oxidised substance in colloidal condition and incapable of penetrating the film. The treatment with oxidised metol, and to a less extent with oxidised quinol, increased both the printing-out speed to daylight and the resulting density of the image. Quinone mixed with quinol has a stronger desensitising action than quinone alone, but mixed with quinol developer or metol-quinol developer green solutions are produced, and there is no desensitising.—B. V. S.

*Negative development by candle light*. Lüppo-Cramer. Der Photograph, 1920, 377–379.

A LARGE number of dyes have been examined as to their action on the sensitiveness of silver bromide, the most active in this respect being dyes of the Safranin class, particularly Phenosafranine, Toluosafranine, the diethyl- and tetramethyl derivatives of Safranin, and Brilliant Rhoduline Red. Developers containing 1/20,000 of Phenosafranine may be used for the development of plates by a bright yellow light, provided they are not exposed to it for the first minute; the process is applicable to colour-sensitised plates, which was not the case with the amidol process (J., 1921, 28 A). The effect is not due to the screening action of the dye solution, since not only is this too weak, but the plate may be lifted from the developer for examination by transmitted light in the usual way. Development is quite normal with metol, metol-quinol, rodinal, amidol, edinol, catechol, pyrogallol, glycin, and quinol, except that the last-named is rendered much more active. By increasing the strength of the dye solution to 1/2000 and using it as a separate bath before development, increased desensitising is obtained along with an added protection due to the staining of the film, and it is then possible to develop any plates, including panchromatic, by un-screened candle light; thorough washing is required to remove the dye from the stained film, assisted, if necessary, by treatment with an acid-alum bath containing 5% of hydrochloric acid and 2% of alum. A desensitising action has also been found with Methylene Blue, Auramine O, and Brilliant Green, but these dyes are not suitable for use on account of fog production or of interference with development. The amino groups of the Safranins appear to be necessary as the replacement of one of them by oxygen (in safraninone) considerably reduces the effect and the replacement of both by hydroxyl (in safranin) almost entirely destroys it.—B. V. S.

*Ives' mordant dye-toning process; New bleach formulae for the —*. F. E. Ives. Brit. J. Phot., 1921, 68, Col. Supplement, 3.

In the bleaching baths previously recommended (J.,

1919, 56a), containing chromic acid with or without potassium ferricyanide, it is an advantage to replace a proportion of the chromic acid by another acid, such as acetic, or hy acetic acid and ammonium bichromate. The hardening action of the bath is reduced, and prolonged wasbing between bleaching and dyeing is avoided.—B. V. S.

*Isocyanine dyes.* Adams and Haller. See IV.

*Dicyanine A.* Mikeska and others. See IV.

*Kryptocyanines.* Adams and Haller. See IV.

#### PATENT.

*Photo-sensitive silver compounds; Process of desensitising* — N. Sulzberger. U.S.P. 1,361,352, 7.12.20. Appl., 10.12.14. Renewed 7.9.20.

*LIGHT-SENSITIVE silver compounds, e.g., in photographic films etc., are desensitised by treatment with a solution of ammonio-palladous chloride.*

—B. V. S.

### XXII.—EXPLOSIVES; MATCHES.

*Nitrations; Decomposition of nitric acid in organic* — F. O. Rice. J. Amer. Chem. Soc., 1920, 42, 2665—2670.

THE following results reproduce the mean of ten experiments on the nitration of phenol under ordinary conditions, varying from the small laboratory scale to a technical operation. The results are calculated on the basis of 100 g. of phenol, 260 g. HNO<sub>3</sub>, and 427 g. H<sub>2</sub>SO<sub>4</sub>. Of the nitric acid 256.78 g. was ac-427 g. H<sub>2</sub>SO<sub>4</sub>. Of the nitric acid 256.78 g. was ac- in the vapours evolved from the nitrating vessel, 9.3 g. evolved as nitrogen peroxide, 17.3 g. as nitric oxide, 36.0 g. as nitrous oxide, 21.78 g. as nitrogen, and 4.6 g. in the spent acid. Of the phenol 94.9 g. was accounted for, viz., 79.2 g. in the picric acid, 5.2 g. oxidised to oxalic acid, 2.4 g. to carbon monoxide, and 8.1 g. to carbon dioxide. By keeping the temperature well below 100° C., especially at the commencement, reduction of the nitric acid to the lowest stages may be avoided, but some oxides of nitrogen are always produced. The best results are obtained by sulphonating almost to the disulphonate stage and then nitrating at about 30°—40° C. (Cf. J.C.S., Feb.)—J. C. W.

*Explosives; Velocity of decomposition of high — in a vacuum. II. Trinitrophenylmethylnitroamine (tetryl).* R. C. Farmer. Chem. Soc. Trans., 1920, 117, 1603—1614.

THE velocity of evolution of gas in a vacuum at 120° C. forms a useful method for the control of the stability of tetryl in the process of manufacture (cf. J., 1921, 63 a), samples of well purified material giving evolutions of 1.5—3.0 c.c. from 5 g. in 40 hrs. The reaction is very sensitive to catalytic influences, and probably one of the main causes of instability is the presence of analogues of tetryl containing a nitro group in the *meta*-position. Traces of picric acid may also be present, and this substance likewise greatly decreases the stability. The temperature coefficient of the decomposition of solid tetryl is 1.9 for 5° C. At the melting point (129° C.) an abrupt change in velocity occurs, the molten substance decomposing about 50 times as rapidly as the solid, and the acceleration in the decomposition at 120° C. is to a great extent due to progressive melting. For a similar reason admixtures which lower the melting point, such as trinitrobenzene or trinitrotoluene, also give rise to a rapid gas evolution. This does not indicate any chemical interaction, and at temperatures below the melting point of the eutectic mixtures the stability does not differ from that of tetryl alone.—G. F. M.

*2,3,6-Trinitrotoluene; Formation of — in the nitration of toluene.* R. B. Drew. Chem. Soc. Trans., 1920, 117, 1615—1618.

THE products of the further nitration of both 2,3- and 3,6-dinitrotoluenes each contain about 15% of 2,3,6-trinitrotoluene, and on this basis it is calculated that crude trinitrotoluene obtained by the direct nitration of toluene contains about 0.3% of this isomeride in addition to the 2,4,6-, 2,3,4-, and 3,4,6-trinitrotoluenes already identified. 2,3,6-Trinitrotoluene was also synthesised from trinitro-*m*-cresol by reducing this with ammonium sulphide to 2,6-dinitro-4-amino-*m*-cresol (methylpicramic acid), eliminating the amino-group, converting the resulting dinitro-*m*-cresol into its methyl ether, replacing the methoxyl group by the amino-group by heating with alcoholic ammonia, and converting the resulting 2,6-dinitro-*m*-toluidine into 2,3,6-trinitrotoluene by Körner and Contardi's method (J., 1915, 1046).—G. F. M.

*Explosives; Identification of — by determining their critical temperature of solution.* L. Crismer. Bull. Soc. Chim. Belg., 1920, 29, 28—33. Chem. Zentr., 1920, 91, IV., 695.

DETERMINATION of the critical temperature of solution provides a simple, rapid, and accurate method of identifying explosives. 2 g. of the substance is heated with 0.9—1.8 c.c. of 95% alcohol till dissolved, and the temperature at which separation occurs on cooling is noted. The critical temperature of solution of  $\alpha$ -mononitronaphthalene is 44° C., and of trinitrotoluene, 96.5° C.—W. J. W.

*Tetranitromethylaniline [tetryl]; Critical temperature of solution of —.* L. Crismer and J. Timmermans. Bull. Soc. Chim. Belg., 1920, 29, 34—35. Chem. Zentr., 1920, 91, IV., 695.

IN a German explosive the presence of tetranitromethylaniline was confirmed by a determination of its critical temperature of solution, which is 105° C.—W. J. W.

#### PATENTS.

*Detonating caps; Manufacture of —.* W. Friederich. E.P. 138,083, 19.1.20. Conv., 27.7.18.

THE composition for the caps consists of a nitro-compound, having above it a charge of lead azide, and above this a charge of lead trinitroresorcinate, either crystallised or dehydrated. Suitable compositions are: tetranitromethylaniline 0.9 g., lead azide 0.2 g., lead trinitroresorcinate 0.2 g.; or nitropentaerythritol 0.7 g., lead azide 0.2 g., lead trinitroresorcinate, 0.2 g. The use of lead trinitroresorcinate ensures a firm surface which will not crumble, and enables the charge to be more easily ignited.—W. J. W.

(A) *Explosive.* (a, c) *Explosive mixture.* R. L. Hill, Assr. to Atlas Powder Co. U.S.P. (a) 1,360,397, (a) 1,360,398, and (c) 1,360,399, 30.11.20. Appl., (a, b) 25.7.19, (c) 30.8.18. All renewed 8.10.20.

AN explosive mixture consists essentially of (c) ammonium perchlorate and nitro-starch, to which (a) an oil, or (b) another diluent ingredient may be added.—W. J. W.

*Smokeless powders; Manufacture of stable —.* C. Claessen. G.P. 298,567, 18.8.15.

NITROCELLULOSE is mixed with more than 21% of a eutectic mixture of solid trinitrotoluene and solid dinitrotoluene, or with complex combinations of these two substances, in presence of a volatile solvent.—W. J. W.

*Explosives; Manufacture of —.* H. Lohmann. G.P. 298,948, 14.1.16.

METALLIC oxides and aluminium, in a fine state of

division and intimately mixed, are compressed, and can then be detonated by means of picric acid, mercury fulminate, dynamite, or guncotton.

—W. J. W.

*Nitration acids from the manufacture of nitrocellulose or the like; Recovery of waste* — S. Hamburger. G.P. (a) 300,758, 3.3.17, and (b) 300,747, 24.5.17.

(A) AFTER removal of the bulk of the waste acid in a centrifuge the residual acid in the nitrocellulose is displaced by spraying in sulphuric acid of approximately the same concentration as the waste acid so as to avoid rise of temperature. To reduce the evolution of nitrous vapours during the removal of the waste acid the centrifuge is run at half speed, but after the sulphuric acid treatment the maximum speed may be attained. (B) The process is similar to that described under (A), but the sulphuric acid is sprayed in at a stage when the nitrocellulose still retains a considerable quantity of nitration acid. (Cf. G.P. 299,680; J., 1921, 29 A.)

—W. J. W.

*Explosive.* R. Mewes. G.P. 302,493, 13.2.17. Addn. to 301,796 (J., 1920, 282 A).

As an absorbent for the compound of oxygen with nitrogen or chlorine, wood or straw, previously digested with alkali and then dried, may be used. Combustible liquids may be added.—W. J. W.

*Drying Explosives; Process for* — C. Gielow and A. Faust. G.P. 325,611, 14.5.18.

By means of a conveyor the explosives are caused slowly to traverse a closed receptacle into which hot air is introduced by means of numerous inlets in the roof and sides. The exhaust air escapes at the rear of the receptacle into a filter where any suspended dust is trapped.—W. J. W.

*Nitric esters; Method of washing* — F. Hoffwimmer. G.P. 325,944, 21.9.18.

AFTER the waste acid has been drawn off, the free acid retained in the nitrated material is neutralised with a saturated solution of an alkali carbonate, in such quantity as to form bicarbonate and avoid evolution of carbon dioxide. A saturated solution of a nitrate may be mixed with the carbonate solution in order to increase the volume of the wash-water and to reduce rise of temperature. The process is applicable to the purification of nitroglycerin and ethyleneglycol nitrate.—W. J. W.

*Explosives; Manufacture of* — in a form in which they can be cast. Sprengstoff A.-G. Carbonit. G.P. 326,184, 7.8.17. Addn. to 307,040 (J., 1920, 282 A).

UREA alone, without a halogen salt, is heated with metallic nitrates or their mixtures. Suitable compositions consist of ammonium nitrate, 80%; sodium nitrate, 10%; urea, 10%; and ammonium nitrate, 90%; urea, 10%.—W. J. W.

*Guncotton and the like; Apparatus for stabilising* — H. Eicheler. G.P. 326,215, 5.10.17.

THE stabilising apparatus consists of an inner and an outer vessel separated from each other. The base of the inner receptacle in which the guncotton is placed is perforated, so that the washing liquid passes through the holes into the intermediate space, which serves as a heating vessel, and circulation of the washing liquid is effected.—W. J. W.

*Illuminating grenade.* J. Sonntag. G.P. (a) 300,760, 15.1.18, (b) 305,099, 15.7.18, and (c) 307,730, 24.4.17.

THE charge in an illuminating grenade consists of (A) magnesium, or (B) aluminium, strontium, barium, or calcium, or of mixtures of these with or without

magnesium. The igniting mixture is composed of barium peroxide and granulated aluminium, or (C) other oxygen carrier mixed with granulated aluminium, magnesium, barium, strontium, or calcium, to which may be added sulphur and a carbonaceous substance, such as charcoal, lampblack, pitch, naphthalene, paraffin, or collodion.

—W. J. W.

*Match-head composition, and method of making same. Composition for match-heads.* W. A. Fairburn, Assr. to The Diamond Match Co. U.S.P. (a) 1,360,282 and (b) 1,360,283, 30.11.20. Appl., 12.8 and 6.10.15.

(A) A match composition contains a mixture of sodium chlorate and zinc dust. (B) A match-head composition contains a mixture of magnetic iron oxide and a metal of which the normal oxide has a greater heat of formation than magnetic iron oxide.

—W. J. W.

### XXIII.—ANALYSIS.

*Dialysis or extraction; Apparatus for continuous* — H. Mann. J. Biol. Chem., 1920, 44, 207—209.

A DISTILLING flask and a dialysing or extracting vessel are connected by two sealed-in tubes; the upper tube is surrounded by a condenser and serves to supply fresh liquid for the operation, while excess liquid is continuously returned to the flask through the lower tube. A bag of collodion etc. is suspended in the dialysing vessel, and means are provided for connecting the apparatus with a vacuum pump.

*Ultra-filters; Simple method of preparing* — E. Knapp-Lenz. Kolloid-Zeits., 1920, 27, 315—316.

AN ultra-filter for use with a Buchner funnel or a Gooch crucible may be prepared as follows:—A 3% solution of collodion is poured into a crystallising dish of the same internal diameter as the Buchner funnel, or into a porcelain crucible of the same internal diameter as the Gooch crucible, and is uniformly distributed by rotating the dish. As soon as the ether has evaporated the dish is filled with water three or four times, and finally the membrane is removed by loosening it at the edges and allowing water to flow between it and the glass. It is then placed in the Buchner funnel, which contains a filter paper, and carefully pressed to the sides of the funnel.—J. F. S.

*Nephelometer; A new — and the principles of nephelometric measurement.* H. Kleinmann. Kolloid-Zeits., 1920, 27, 236—241.

A NEW type of nephelometer is described, which depends on the measurement of the height of a Tyndall cone which produces the same intensity of illumination in two liquids, one of which is a standard. The apparatus consists of two glass tubes each graduated to a suitable volume, and containing the liquids for comparison. These tubes are placed side by side on a metal table before two shutters which may be opened to any measured amount. Into the glass tubes, two cylinders of polished glass just dip, and the light passing through these from the Tyndall cones illuminates the two halves of an optical system. The width of the shutter opening is then regulated until the two halves of the field are equally illuminated. The height of the Tyndall cone is controlled by the width of the opening of the shutters, and the intensity of illumination varies directly with the concentration. Experiments show that there is a strict proportionality between turbidity and concentration in the two tubes for the ratio 1:4 between the concentration in the two tubes. The instrument is easy



to operate, and amounts of phosphoric acid ( $P_2O_5$ ) down to 0.0005 mg. in 25 c.c. of solution may be estimated with it (cf. J., 1920, 707 A) with an average error of 0.5%. An essential point is that the size of the particles in the turbid liquid is uniform.

—J. F. S.

*Gas analysis by absorption and titration.* R. S. Tonr. Chem. and Met. Eng., 1920, 23, 1104—1106.

For determining the volume of residual gas, after absorption of one constituent by a suitable reagent, a gas volume compensometer is employed. It comprises a gas flask, having a two-way tap at one end, by means of which communication may be established either with the absorption vessel or with one limb of a manometer. The other limb of the manometer is connected with the compensometer, which is a closed tube or bulb completely enclosed by the gas flask. At the end opposite to the two-way tap the flask is connected with a water reservoir. After filling the flask with water, the residual gas is drawn in by putting the flask in communication with the absorption vessel and running out water from the flask. The tap is then turned to connect the flask with the manometer, on the scale of which either the correction factor or the corrected volume (at 760 mm.,  $0^\circ C.$ , dry) may be read off. The percentage of the constituent, Y, in the original gas is determined from the formula  $(100-Y)/Y = VJ/22.4NQ$ , where V=vol. of residual gas corrected to standard conditions, J=valency of absorbed constituent, N=normality of reagent, and Q=c.c. of reagent used for titration. The methods of calibrating the compensometer and of graduating the manometer scale are indicated, and a nomograph for the graphical solution of the above equation is described.—W. J. W.

*Hydrogen ion concentration; Determination of the — by means of indicators.* L. Michaelis and A. Geymant. Biochem. Zeits., 1920, 109, 165—210.

On adding a solution of a certain hydrogen ion concentration to an indicator a coloration of a definite depth is obtained, and by estimating colorimetrically the depth of the colour produced it is possible to determine the hydrogen ion concentration. The best conditions under which these estimations can be carried out are described, and the formula for calculating the results is given.—S. S. Z.

*Barium and strontium; Volumetric determination of — as chromates.* I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 972—979.

Barium and strontium respectively can be quantitatively precipitated as chromates by addition of dichromate under suitable conditions, and the excess of chromate may be estimated iodometrically in an aliquot part of the filtrate. Barium can be accurately determined in the presence of strontium or calcium, but strontium is not quantitatively precipitated as chromate from solutions containing barium or calcium. (Cf. J.C.S., 1921, ii., 62.)

—S. I. L.

*Nitrogen; Micro-determination of — in agricultural materials.* W. Geilmann. J. Landw., 1920, 68, 235—249.

A method for the determination of nitrogen in very small quantities of such substances as proteins, ammonium salts, etc., is described. It consists essentially of a micro-Kjeldahl digestion, the ammonia formed being distilled subsequently in a current of steam or removed by a current of air, absorbed in a definite quantity of standard acid, and the excess of this then titrated. Directions are given for the preparation of the reagents used, means for preventing atmospheric contamination, etc.—W. P. S.

*Kjeldahl method for determining nitrogen; Investigation of the —.* I. K. Phelps and H. W. Dandl. J. Assoc. Off. Agric. Chem., 1920, 4, 72—76.

THE results confirm those given previously (J., 1920, 351 A). Using the proportions given there, the 10 g. of potassium sulphate may be replaced by 8.2 g. of sodium sulphate. The influence of the reagents and the apparatus on the accuracy of the modified Kjeldahl process is indicated, and the desirability of control by blank experiments in routine work is emphasised.—W. G.

*Kjeldahl method modified for nitrates; Use of permanganate in the —.* I. K. Phelps. J. Assoc. Off. Agric. Chem., 1920, 4, 69—71.

IN the estimation of nitrates by the modified Kjeldahl method using a mixture of sulphuric acid and salicylic acid, the addition of potassium permanganate at the end of the boiling may cause the loss of nitrogen and it should therefore be omitted.

—W. G.

*Nitrogen determinations; Influence of potassium permanganate on Kjeldahl —.* D. C. Cochran. J. Ind. Eng. Chem., 1920, 12, 1195—1196.

THE addition of permanganate to the hot acid digestion mixture did not cause loss of nitrogen, provided that the addition was made within 2 mins. after cessation of boiling; the results obtained for feeding-stuffs and faeces are more concordant and uniformly higher when permanganate is used.

—W. P. S.

*Amino-nitrogen; Iodometric determination of — in organic substances.* H. H. Willard and W. E. Cake. J. Amer. Chem. Soc., 1920, 42, 2646—2650.

THE Kjeldahl estimation can be shortened by cooling the mixture as soon as the organic matter is thoroughly charred, adding dry potassium persulphate, warming carefully until the liquid is colourless, which requires about 1 min., and then boiling to decompose the excess of persulphate. The solution is then cooled, rendered just alkaline, and the ammonia is estimated by adding an excess of standard hypobromite, leaving for a few minutes, then adding potassium iodide and hydrochloric acid and titrating with thiosulphate.—J. C. W.

*Methoxyl group; Volumetric determination of the —.* J. Troeger and E. T. Tiebe. Arch. Pharm., 1920, 258, 277—287.

THE quantitative demethylation of cusparine by dry hydrogen chloride (Troeger and Müller, J., 1915, 449) suggested the use of the method for the determination of methoxyl. Essentially the process consists in heating the material (0.1 g.) in a stream of dry hydrogen chloride, the temperature being gradually raised until bubbles of gas are seen to escape from the substance. The methyl chloride so produced is collected in a nitrometer over 30—35% sodium hydroxide solution. The results obtained in a number of cases were not absolutely accurate, but sufficed as a clear indication of the number of methoxyl groups present. The Zeisel method is not applicable to compounds containing sulphur, but the present method gave an approximate value in the case of galipinsulphonic acid, although not in another instance. (Cf. J.C.S., Feb.)—J. K.

*Phosphatides; Quantitative determination of —.* D. H. Brauns and J. A. MacLaughlin. J. Amer. Chem. Soc., 1920, 42, 2238—2250.

ANIMAL or vegetable tissues which cannot be reduced to powder, or solutions in volatile liquids, are dried on a water bath with a mixture of anhydrous sodium and calcium sulphates (cf. Robertson, J. Biol. Chem., 1916, 24, 409). The dry material is extracted



with absolute alcohol at 50°–60° C. in a Soxhlet apparatus. The extract is evaporated to dryness and extracted with dry ether. The ethereal extract is evaporated to 30–40 c.c., shaken with 15 c.c. of a saturated solution of sodium chloride, and then separated and evaporated to dryness and the residue weighed. In this residue the phosphorus is estimated colorimetrically by Van Eck's method (J., 1918, 559A) by reduction with stannous chloride, the organic matter being first destroyed by Nenmann's method of heating with sulphuric and nitric acids (Z. physiol. Chem., 1902, 37, 130). As a check on the phosphorus estimation, choline is estimated as its platinichloride after acid hydrolysis and amino-nitrogen is estimated by Van Slyke's method (cf. J., 1911, 771). From the results for choline and amino-nitrogen the ratio of lecithin to kephalin is established.—W. G.

See also pages (A) 77; *Nitrogen oxides* (Taylor); 78, *Phosphates* (Hillebrand and Lundell); 79, *Graphite* (Taylor and Selvig); 89, *Spermaceti* (Bohrisch and Kürschner); 92, *Dicyanodiamide* (Harger); 93, *Oxidation of dextrose* (Witzemann); 94, *Fat in skimmed milk* (McInerney and Troy); *Proteins* (Holm and Gortner); 95, *Turbidity standard* (Wells), *Carbonic acid in water* (Koltzoff); 98, *Thymol* (Mastbaum); 100, *Explosives* (Crismer).

#### PATENTS.

*Calorimeters.* Soc. des Hauts-Fourneaux de Rouen. E.P. 145,437, 18.6.20. Conv., 10.5.19.

THE combustible fluid is supplied at a constant rate to a burner placed within a chamber having conducting walls, e.g., a hollow thick-walled copper cylinder, surrounded by a jacket and provided with means for supplying primary and secondary air for combustion. Means are provided for measuring or recording the temperature of the walls of the chamber, which serves as a measure of the calorific value of the combustible fluid.—J. S. G. T.

*Combustible gases; Method of and means for determining the calorific value of* —. Igranic Electric Co., Ltd. From The Cutler Hammer Manufacturing Co. E.P. 153,817, 4.2.20.

Gas and air are supplied to a mixing chamber in constant proportions by two wet displacement meters geared together and immersed in a tank so that the gases are under similar conditions of temperature, pressure, and saturation. The mixed gas passes to a burner enclosed in a tube closed at the top, so that the products of combustion are forced to travel downwards through this tube. The heat of combustion is absorbed by a stream of air which passes upwards through an outer concentric tube, the air being in a definite proportion determined by passing it through a third meter geared to the other two meters. If the proportions are kept constant, the temperature of the last-mentioned stream of air gives a measure of the calorific value of the gas. The readings are taken by electric resistance thermometers, and means are provided for compensating for variations of the calorific value due to dilution of the gas with water vapour, variations in the specific heat of the cooling gas with temperature, and incomplete transfer of heat to the cooling gas.—W. F. F.

*Analysis of gases; Apparatus for the continuous* —. T. R. Wollaston. E.P. 153,637, 23.7.19.

A SYMMETRICAL double acting pump delivers the gas into two graduated vessels filled with inert liquid. The pipe connexions are such that an absorption vessel may be placed in circuit between the pump and either receiver, or omitted from both circuits.

The liquid displaced from each receiver in a given period is continuously measured or weighed by a recording apparatus, and if one receiver includes the absorption vessel in its circuit while the other does not, the difference in the readings gives the amount of gas absorbed.—W. F. F.

*Gas-analysing apparatus; Automatic* —. O. Rodhe, Assr. to Svenska Aktiebolaget Mono. U.S.P. 1,351,129, 31.8.20. Appl., 26.1.20.

An automatic gas-analysing apparatus includes the usual primary measuring vessel, absorption vessel, and secondary measuring vessel. A branch pipe including a small additional measuring vessel connects the primary measuring vessel with the pipe connecting the absorption vessel with the secondary measuring vessel. This small additional vessel thus contains neutral gas, which is alternately forced into the absorbing vessel and into the secondary measuring vessel. This gas supplies the pressure necessary to effect a measurement in the second measuring vessel, and thus enables readings to be obtained up to 100% absorption.—W. F. F.

*Quantitative analysis of colour; Instrument for the* —. A. E. Bawtree. E.P. 154,671, 25.8.19.

THE operation of the instrument depends upon the principle that any shade of colour may be obtained by mixture in suitable proportions of two or more of the primary colours, scarlet, green, and violet-blue. Light from a single source is divided into two parts, one of which illuminates the sample to be tested while the other passes through three or more adjacent but not superimposed coloured screens or filters, the coloured beams thus produced being subsequently combined. A calibrated device is provided for regulating the quantity of white light illuminating the sample and for determining the relative intensities of the coloured beams transmitted by the three coloured screens when the colour afforded by the combined colours matches the colour of the sample. For the examination more especially of opaque objects, a slotted reflector is provided in the path of the coloured beam, enabling the sample illuminated by white light to be viewed through the slot.—J. S. G. T.

*Carbon, hydrogen, and nitrogen determinations; New micro-combustion furnace for* —. W. Dautwitz. Chem.-Zeit., 1920, 44, 963.

THE furnace, which can be used for ordinary combustions or for nitrogen determinations, is mounted, together with absorption stands, on a single board for convenience in moving. The combustion tube is heated from the sides by gas jets from two parallel gas tubes fed from three Bunsen burners which are fixed to the board and adapted as supports for the furnace. (Cf. J.C.S., Feb.)—J. H. L.

*Moisture in grain.* E.P. 151,315. See XIXA.

#### Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

#### I.—GENERAL; PLANT; MACHINERY.

##### APPLICATIONS.

American Coke and Chemical Co. By-product condensers. 1553. Jan. 10. (U.S., 17. and 15.)  
Brown. Autoclaves. 2531. Jan. 19.

Cartner, Clewer, and Mather and Platt. Vacuum filtration apparatus. 2799. Jan. 21.  
 Catlin Shale Products Co. Decolorising-materials. 1314. Jan. 10. (U.S., 25.4.16.)  
 Chem. Fabr. Worms. Distillation. 1575. Jan. 10. (Ger., 6.8.17.)  
 Dingers'che Maschinenfabr. A.-G. Purifying gases and vapours. 1532. Jan. 10. (Ger., 1.9.13.)  
 Gerken. Furnaces. 1408-1410. Jan. 10.  
 Griffith. Grinding-machines. 1551. Jan. 10. (U.S., 18.4.18.)  
 Hernu. Apparatus for purifying and treating gases. 1198. Jan. 10. (Fr., 9.12.18.)  
 Hodge and others. 1946. *See* VII.  
 Hulsmeyer. Separating air and gases from boiler feed-water etc. 1505-6. Jan. 10. (Ger., 11.11.13 and 11.10.15.)  
 Kestner. Abstracting gases from water by metallic filters. 1399, 1400. Jan. 10. (Fr., 10.6 and 22.7.20.)  
 Kestner. Distillation of water. 1402. Jan. 10. (Fr., 15.6.20.)  
 Kestner. Metallic media for fixing oxygen of water in apparatus for abstracting gases from liquids. 2275. Jan. 17. (Fr., 9.6.20.)  
 Kestner. Heating and decanting apparatus for purifying feed-water. 2817. Jan. 21. (Fr., 12.6.20.)  
 Krause. Mixing granular substances. 1976. Jan. 13.  
 Lundie. Filtering apparatus. 2070. Jan. 14.  
 Mather. Pre-heaters, heat-exchangers, condensers, etc. 1786. Jan. 11.  
 Reid. Furnaces. 2448. Jan. 18.  
 Rialland. Centrifugal separator for clarifying liquids. 1746. Jan. 11. (Fr., 30.4.19.)  
 Rippl. 1528. *See* II.  
 Salenius. Centrifugal separators. 1248-1250. 1253-5, 1259-1262. Jan. 10.  
 Vaccaro. Desiccators. 2581. Jan. 19.  
 Vernon. Tunnel ovens or kilns. 2036. Jan. 14.  
 Wade (Schneible). Distilling. 2003. Jan. 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

3701 (1918). Paterson. Filtering apparatus. (156,270.) Jan. 19.  
 6069 (1919). Kirke. Boiler furnaces. (156,826.) Jan. 26.  
 15,543 and 32,529 (1919). Kermode. Furnaces. (156,834.) Jan. 26.  
 16,148 (1919). Darneden. Rotary furnaces. (156,835.) Jan. 26.  
 21,188 (1919). McLaughlin. Pulverising apparatus. (156,852.) Jan. 26.  
 24,516 (1919). Steinmuller. Travelling-grate furnaces. (133,691.) Jan. 19.  
 24,936 (1919). Dorr Co. Sedimentation apparatus. (133,716.) Jan. 26.  
 26,543 (1919). Gardiner and Storey. Furnaces. (156,364.) Jan. 19.  
 28,278 (1919). South Metropolitan Gas Co., and Parrish. *See* VII.  
 31,661 (1919). Soc. Milanese Impianti Ind. C. Crespi-Squassi. Rotary pulveriser. (136,831.) Jan. 19.  
 3496 (1920). Barron. *See* XIX.

5227 (1920). Kennedy. Crnshing and pulverising machines. (139,219.) Jan. 19.  
 18,203 and 18,396 (1920). Chem. Fabr. Weilerter Meer. Centrifugal pumps for acids. (145,803 and 146,409.) Jan. 26.

#### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

##### APPLICATIONS.

Act.-Ges. f. Anilinfabr. Fuel for internal-combustion engines. 1589. Jan. 10. (Ger., 8.11.19.)  
 American Coke and Chemical Co. Reflex coke oven. 1552. Jan. 10. (U.S., 22.6.16.)  
 American Coke and Chemical Co. 1553. *See* I.  
 Bates. Treating fuels. 2115. Jan. 14. U.S., 6.2.20.)  
 Bates. Production of gas. 2212. Jan. 15. (U.S., 19.2.20.)  
 Boardman and Madden. Steaming and aeration of vertical retorts. 1810. Jan. 12.  
 Brooke and Whitworth. Apparatus for manufacture of gas. 1984. Jan. 13.  
 Brooke and Whitworth. Furnaces for making producer-gas etc. 2393. Jan. 18.  
 Catlin Shale Products Co. 1314. *See* I.  
 Dickson. Gas-producers. 1774. Jan. 11.  
 Dolensky. Gas-producers. 1587. Jan. 10. (Ger., 22.2.17.)  
 Du Pont de Nemours and Co. 1322. *See* XII.  
 Foster. 1690. *See* IX.  
 General Oil Gas Corp. Gas manufacture. 2580. Jan. 19. (U.S., 9.8.20.)  
 Grosse. Gas etc. purification. 1682. Jan. 10. (Ger., 1.9.13.)  
 Halbergerhütte Ges. 2310, 2696-7. *See* X.  
 Illing and Kelly. Production of asphalt from petroleum etc. 2833. Jan. 21.  
 Jacobs. Improvement of inferior brown coals and peat. 1510. Jan. 10. (Ger., 28.7.19.)  
 Jacobs. Production of gas-coal substitute. 1511. Jan. 10. (Ger., 28.7.19.)  
 Jacobs. Recovery of methane. 1778. Jan. 11. (Ger., 12.1.20.)  
 Mather. Fractional distillation of crude oil etc. 1785. Jan. 11.  
 Nielsen. Plant for distilling carbonaceous materials. 2120. Jan. 14.  
 Oddy. Distilling coal, peat, wood, etc. 2614. Jan. 20.  
 Pollacsek. Manufacture of briquettes. 1609. Jan. 10. (Austria, 17.10.19.)  
 Polysius. Low-temperature carbonisation. 1230. Jan. 10. (Ger., 1.2.18.)  
 Raschig. 1509. *See* III.  
 Rigby. Drying peat etc. 2372. Jan. 18.  
 Rippa. Continuous distillation or gasification of organic matter. 1528. Jan. 10. (Ger., 25.8.15.)  
 Salenius. Drying peat. 1256. Jan. 10. (Sweden, 4.5.17.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

14,750 (1916). Helps. Gas manufacture. (156,812.) Jan. 26.

17,114 (1919). Dunstan and Thole. Treatment of heavy oils. (156,234.) Jan. 19.

29,010 (1919). Stevens. Motor fuels. (135,514.) Jan. 26.

30,930 (1919). Hoover and Brown. Treatment of shale. (156,396.) Jan. 19.

6932 (1920). Matsunaga and Aoki. Manufacture of fuel briquettes. (157,007.) Jan. 26.

18,076 (1920). Dempster, Ltd., Knight, and Siddall. Coal or fuel carbonising and gas-making plant. (157,036.) Jan. 26.

19,126 (1920). Still. Treating crude gas liquor to obtain ammonia solution. (147,099.) Jan. 19.

### III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Andrews, Conover, John, and Ruth. Purification of naphthalene. 2554. Jan. 19.

American Coke and Chemical Co. 1553. *See* I.

Mauthner and Pfeifer. Production of halogen derivatives from hydrocarbons. 1146. Jan. 10. (Hungary, 20.11.18.)

Otto. Treatment of tar oils etc. 2297. Jan. 17. (Ger., 17.1.20.)

Raschig. Recovery of benzol hydrocarbons from coke-oven gas. 1509. Jan. 10. (Ger., 23.11.13.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

2366 (1918). Steele and Robertson. Conducting fusions with molten caustic soda or caustic potash. (156,269.) Jan. 19.

15,454 (1920). Kinzberger u. Co. Purification of crude anthracene. (144,656.) Jan. 26.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

Davies, and Scottish Dyes, Ltd. Production of colouring matters of the anthraquinone series. 1991. Jan. 13.

Lüers. 1593. *See* XVIII.

Phillips. Artificial dyes. 2493. Jan. 19.

#### COMPLETE SPECIFICATION ACCEPTED.

14,767 (1920). Inray (Monsanto Chemical Works). Manufacture of phthaleins. (157,030.) Jan. 26.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

British Cellulose and Chem. Manuf. Co., and Dickie. Coating processes and apparatus. 2694. Jan. 20.

British Cellulose and Chem. Manuf. Co., and Addy. Manufacture of articles of plastic materials. 2695. Jan. 20.

Budde, and Hendon Paper Works Co. Substitute for celluloid. 2109. Jan. 14.

Clavel. Treatment of cellulose acetate. 1911. Jan. 12.

Dreaper. Manufacture of artificial silk etc. 2598. Jan. 19.

Gierisch and Waentig. Obtaining cellulose. 1562. Jan. 10.

Gierisch, Waentig, and Kraus. Production of single fibres from bast fibre bundles, with simultaneous disintegration and removal of wood particles etc. 1566. Jan. 10. (Ger., 4.4.19.)

Kämpf. Apparatus for reducing alkali cellulose etc. 1832. Jan. 12. (Ger., 13.1.20.)

McKellar. Treatment of textile fibres to remove grease, wax, etc. 1968. Jan. 13.

Pollacsek. Manufacture of mastic or binding substance from sulphite lye. 1608. Jan. 10. (Austria, 17.10.19.)

Sabner. Degumming etc. textile fibres. 2680. Jan. 20.

Salmon. Bleaching fibres in manufacture of paper. 2099. Jan. 14.

Soc. Anon. la Cellophane. Bands of cellulosic material. 2587. Jan. 19. (Fr., 4.3.20.)

Soc. Anon. la Cellophane. Straw, hair, etc., manufactured from cellulose material. 2588. Jan. 19. (Fr., 4.3.20.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

16,617 (1919). Picard. Extraction of fibres from flax straws or waste. (156,281.) Jan. 19.

16,861 (1920). Badische Anilin- u. Soda-Fabrik. Manufacture of solutions of cellulose acetate. (145,511.) Jan. 26.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### APPLICATIONS.

Crompton. Treatment of printed fabrics. 2732. Jan. 21.

Herron and McKerrow. Production of ornamental effects on silk etc. 1881. Jan. 12.

Krantz. Means for dyeing yarn on bobbins. 1343—6. Jan. 10. (Ger., 4.10 and 12.11.13, 12.1.14, 17.1.17.)

Krantz. Drying textile materials. 1350. Jan. 10. (Ger., 15.1.17.)

Morgan. Dyeing-machines. 2418. Jan. 18.

Pollak. Production of stiffening agents. 1341. Jan. 10. (Ger., 6.11.19.)

Salmon. 2099. *See* V.

#### COMPLETE SPECIFICATIONS ACCEPTED.

23,232 (1919). Walsh and Tomlinson. Apparatus for printing and sizing yards etc. (156,302.) Jan. 19.

25,437 (1919). Calico Printers' Assoc., and Costabadie. Treatment of cloth. (156,913.) Jan. 26.

26,227 (1919). Hunt. Apparatus for bleaching treatment of open fabrics. (156,359.) Jan. 19.

### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

#### APPLICATIONS.

Brat. Recovery of ammonia from peat. 1447—8. Jan. 10. (Ger., 21.10 and 20.7.18.)

British Cellulose and Chem. Manuf. Co., and Bader. Manufacture of pyrosulphates. 2693. Jan. 20.

Delarozière. Manufacture of sodium and potassium ferrocyanide. 2224. Jan. 17.

Deuts.-Luxemburgische Bergwerks- u. Hütten-A.-G., and Hilpert. Treatment of waste sulphuric acids. 1415. Jan. 10. (Ger., 12.5.16.)

Foster. 1690. *See* IX.

Helbronner and Pipereaut. Manufacture of sulphuric acid. 1191. Jan. 10. (Fr., 29.4.18.)

Hodge, and Ovens and Sons. Sulphuric acid valves. 1946. Jan. 13.

Lidholm and others. 2411. *See* XVI.

Lundén and Thorssell. Production of nitrogen compounds. 2589. Jan. 19.

Lundén and Thorssell. Preparation of a mixture of compounds of alkaline-earth metals and carbon for absorption of nitrogen. 2914. Jan. 22.

New Jersey Zinc Co. 2104 and 2119. *See* XIII.

Parrish and others. 1790. *See* X.

Pearson. 2281. *See* XIII.

Wallin. 1637. *See* IX.

#### COMPLETE SPECIFICATIONS ACCEPTED.

24,386 (1919). South Metropolitan Gas Co., and Parrish. Manufacture of sulphuric acid. (156,328.) Jan. 19.

24,925 (1919). Wade (Lindsay Light Co.). Manufacture of insoluble thorium compounds. (156,892.) Jan. 26.

25,479 (1919). Dreyfus. Manufacture of acetic acid. (156,916.) Jan. 26.

28,278 (1919). South Metropolitan Gas Co., and Parrish. Means for drying salts. (156,963.) Jan. 26.

32,414 (1919). Toniolo, and Officine Elettrochim. Dr. Rossi. Production of hydrogen and mixtures of hydrogen and nitrogen. (152,975.) Jan. 19.

19,126 (1920). Still. *See* II.

19,675 (1920). Bea. Manufacture of copper sulphate. (147,689.) Jan. 26.

#### VIII.—GLASS; CERAMICS.

##### APPLICATIONS.

Koppens. Tunnel kilns for pottery, lime-burning, etc. 1529. Jan. 10.

Paziczky. Producing spun glass. 1277. Jan. 10. (Ger., 7.4.19.)

Sutton. Colouring or treating glass etc. 2045. Jan. 14.

Vernon. 2036. *See* I.

#### COMPLETE SPECIFICATIONS ACCEPTED.

11,002 (1920). Rollason. Manufacture of refractory materials for furnace linings. (156,447.) Jan. 19.

26,523 (1920). Rheinberg. Producing reflecting surfaces on glass by means of metals of the platinum group. (156,472.) Jan. 19.

#### IX.—BUILDING MATERIALS.

##### APPLICATIONS.

Berry. Manufacture of artificial stone, cement, etc. 2319. Jan. 17.

Foster. Production of lime for building, together with ammonia and a gas for power purposes. 1690. Jan. 11.

Benyi and Renyi. Bricks etc. 1356. Jan. 10. (Austria, 10.1.14.)

Renyi and Renyi. Building materials for use as wood substitutes. 1355 and 1358. Jan. 10. (Austria, 10.1 and 9.5.14.)

Wallin. Production of steinholz from magnesia and magnesium salts. 1637. Jan. 10. (Ger., 12.3.14.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

18,844 (1918). Atkins and Colquhoun. Manufacture of bricks, clinker, etc. (156,820.) Jan. 26.

7901 and 11,042 (1920). Fabr. Akt. Kronstein. Insulating and building material. (140,431 and 142,111.) Jan. 19.

9786 (1920). Forrester (Internat. Isolations Komp.). Manufacture of porous stones for thermal insulating purposes. (156,442.) Jan. 19.

10,435 (1920). Holzbearbeitungs Ges., Haring, and Voit. Drying wood. (141,727.) Jan. 26.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

##### APPLICATIONS.

Ashcroft. Metallurgy of lead and zinc and their sulphides. 2068. Jan. 14.

Ballantine. Manufacture of ferro chromium alloys. 2592. Jan. 26.

Basset. Direct production of iron, steel, iron alloys, etc. 1318-9. Jan. 10. (Fr., 2.10.18, 20.12.17.)

Bishop. Removing metallic constituents from a mixture thereof. 1890. Jan. 12. (U.S., 14.1.20.)

British Thomson-Houston Co. Platinum alloys. 1920. Jan. 12. (U.S., 5.3.14.)

Cliff. Treating scrap iron. 2585. Jan. 19.

Corsalli. Enriching iron alloys poor in silicon and manganese. 1655 and 1658. Jan. 10. (Ger., 27.6 and 27.7.17.)

Corsalli. Smelting iron and steel scrap. 1656. Jan. 10. (Ger., 8.5.18.)

Corsalli. Smelting. 1657, 1659, 1660. Jan. 10. (Ger., 2.6.16., 18.9.13, 20.3.18.)

Corsalli. Furnaces. 1661. Jan. 10. (Ger., 6.5.19.)

Davies. Steel or iron nickel welding. 1824 and 1826. Jan. 12.

Diepschlag. Regulating working of waste gases in blast furnace operations. 1757. Jan. 11. (Ger., 30.6.20.)

Dunkley and Ryan. White metal alloy. 2569. Jan. 19.

Halbergerhütte Ges. Purification of blast-furnace etc. gases. 2310 and 2696. Jan. 17 and 20. (Ger., 1.12.20.)

Halbergerhütte Ges. Air and gas purifiers. 2311 and 2697. Jan. 17 and 20. (Ger., 1.12.20.)

Kuhasta. Heat treatment of steel. 1194. Jan. 10.

Linnmann. Production of raw or cast iron clippings. 1206. Jan. 10. (Ger., 28.4.19.)

Lohmann. Manufacture of blocks from wolfram, molybdenum, etc. 1449, 1452-3. Jan. 10. (Ger., 2 and 29.1 and 16.4.14.)

Lohmann. Manufacture of hard but ductile tools. 1475. Jan. 10. (Ger., 7.12.18.)

Lohmann. Manufacture of hard alloys. 1480. Jan. 10. (Ger., 7.12.18.)

Lohmann. Withdrawing carbon from alloys of high melting temperatures. 1488. Jan. 10. (Ger., 13.1.19.)

Loke. Manufacture of ferrous titanium or refined iron or steel. 1404. Jan. 10.

Parrish, Weight, and South Metropolitan Gas Co. Treatment of waste liquor from wet extraction of copper. 1790. Jan. 11.

Platt. Repairing aluminium sheet and castings and attaching copper, brass, steel, etc. thereto. 2849. Jan. 22.

Slatineanu. Electrolytic separation of platinum from other metals. 1500. Jan. 10. (Switz., 19.12.19.)

Volmer. Production of copper coatings on non-metallic materials. 1300. Jan. -0. (Ger., 12.11.19.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

23,960 (1919). Ashcroft. Treatment of ores or other metal-bearing materials. (156,866.) Jan. 26.

4864 (1920). Levoz. Manufacture of soft cast-iron. (140,756.) Jan. 26.

9651 (1920). Metropolitan-Vickers Electrical Co. Improving the magnetic qualities of magnetisable materials. (141,345.) Jan. 26.

26,523 (1920). Rheinberg. See VIII.

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Adam and Antonoff. Electric batteries. 1592. Jan. 10.

British Thomson-Houston Co. (General Electric Co.). Electric furnaces. 2417. Jan. 18.

British Thomson-Houston Co. (General Electric Co.). Electrodes, and methods of making same. 2911. Jan. 22.

Cattley. Accumulator plates. 1291. Jan. 10.

Electrosynthese Ges. Carrying out electro-chemical gas reactions. 2269. Jan. 17. (Austria, 19.7.17.)

Juchli. Production of electric insulating material. 1340. Jan. 10. (Switz., 8.8.19.)

Rankin, and Van Raden and Co. Electric accumulators. 2899. Jan. 22.

Slatineanu. 1500. See X.

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,821 (1919). Akt. Kfäfveindustri. Heating material or performing chemical processes in electric furnaces. (141,656.) Jan. 26.

7901 and 11,042 (1920). Fabr. Akt. Kronstein. See IX.

9686 (1920). Assie. Electric furnaces. (141,352.) Jan. 26.

15,775 (1920). Elektro-Osmose A.-G. Electro-osmotic separation of substances. (144,710.) Jan. 19.

### XII.—FATS; OILS; WAXES.

#### APPLICATIONS.

Chadbonrne. Manufacture of soap. 1460. Jan. 10.

Du Pont de Nemours and Co. Removing odours from treated oils. 1322. Jan. 10. (U.S., 13.6.18.)

Faester. Recovering oil from fish liver etc. 1547. Jan. 10. (Norway, 15.10.19.)

Gonyers, Reynard, and Lanoline Extractors, Ltd. Treatment of wool fats etc. 2917. Jan. 22.

Wright. Manufacture of margarine etc. 2633. Jan. 20.

#### COMPLETE SPECIFICATION ACCEPTED.

25,213 (1919). Bollmann. Extraction of fat or oil from raw materials. (156,905.) Jan. 26.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

Clerc and Nihoul. Manufacture of zinc white. 1538. Jan. 10. (Fr., 15.11.19.)

Koller. Manufacture of synthetic resins. 2065. Jan. 14.

New Jersey Zinc Co. Manufacture of French oxide. 2104. Jan. 10. (U.S., 31.3.20.)

New Jersey Zinc Co. Manufacture of zinc oxide. 2119. Jan. 10. (U.S., 28.6.20.)

Pearson. Manufacture of zinc oxide. 2281. Jan. 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

9433 (1919). Lillie. Poison compositions for use as antifouling paints etc. (156,827.) Jan. 26.

24,978 (1919). Howse. Solutions of phenol-aldehyde condensation products. (156,896.) Jan. 26.

29,417 (1919). Nishizawa. Sulphide composition for painting which resists the action of light and heat. (156,971.) Jan. 26.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### APPLICATIONS.

Hopkinson. Products obtained from rubber-containing latex. 1769. Jan. 11. (U.S., 16.1.20.)

Hopkinson. Treatment of rubber-containing latex. 1782. Jan. 11. (U.S., 16.1.20.)

Western Rubber Co. Rubber substitute. 1561. Jan. 10. (U.S., 16.3.18.)

### XV.—LEATHER; BONE; HORN; GLUE.

#### APPLICATIONS.

Burger. Tawing and currying leather. 1640. Jan. 10. (Ger., 14.5.14.)

Chem. Fabr. Worms. Tanning hides. 1577. Jan. 10. (Ger., 6.8.17.)

Chem. Fabr. Worms. Manufacture of tanning agents. 1578. Jan. 10. (Ger., 31.5.16.)

Chem. Fabr. u. Asphaltwerke. Manufacture of tanning agents and process of tanning therewith. 1583-4. Jan. 10. (Ger., 21.6 and 17.7.16.)

Lamplough, and Townmead Construction Co. Material for use as a binder. 2467. Jan. 18.

Reubig. Clearing, removing colour from, and finishing leathers. 1595. Jan. 10. (Ger., 25.7.16.)

#### COMPLETE SPECIFICATION ACCEPTED.

10,445 (1920). Jackson (Dorr Co.). Recovery of proteids from waste liquors of the hide-treating art. (156,444.) Jan. 19.

### XVI.—SOILS; FERTILISERS.

#### APPLICATION.

Lidholm, and Wargöns Aktiebol. Producing cyanamide from calcium cyanamide. 2411. Jan. 18. (Sweden, 3.3.20.)

## COMPLETE SPECIFICATION ACCEPTED.

12,571 (1920). Hamler. Fertiliser dryers. (143,846.) Jan. 26.

## XVII.—SUGARS; STARCHES; GUMS.

## APPLICATIONS.

Campbell. Manufacture of starch. 1800. Jan. 11.  
Kowalski. Obtaining sugar from extractions and molasses. 2296. Jan. 17.

## COMPLETE SPECIFICATION ACCEPTED.

22,774 (1919). Kowalski. Manufacture of a precipitate rich in albumen and poor in ash from raw juices and waste liquors of sugar factories. (132,798.) Jan. 19.

## XVIII.—FERMENTATION INDUSTRIES.

## APPLICATIONS.

Bevan. Siphons for making beer etc. 2738—9. Jan. 21.  
Bevan. Vats for fermenting beer etc. 2740. Jan. 21.  
Lüers. Production of colouring-medium for beers etc. 1593. Jan. 10. (Ger., 17,12,19.)

## XIX.—FOODS; WATER PURIFICATION; SANITATION.

## APPLICATIONS.

Bartmann. Producing flour from corn. 2821. Jan. 21.  
Berczeller and Graham. Synthetic milk. 1264—5. Jan. 10. (Austria, 26,7,19, 7,1,20.)  
Candy Filter Co. Destruction of micro-organisms in liquids. 1190. Jan. 10. (Austria, 8,1,20.)  
Cholet. Preserving meat, fish, etc. 2692. Jan. 20.  
Daw. Treatment of sewage etc. 2295. Jan. 17.  
Fitzgerald. Preparation of industrial substances from blood. 2268. Jan. 17.  
Hulsmeyer. 1505—6. See I.  
Kestner. 1399, 1400, 1402, 2275, 2317. See I.  
Martin and Wallis. Manufacture of condensed milk. 1906. Jan. 12.  
O'Loughlin. Manufacture of feeding material for animals. 1828. Jan. 12.  
Prokop. Treatment of cereals. 2009. Jan. 13.  
Wright. 2633. See XII.

## COMPLETE SPECIFICATIONS ACCEPTED.

11,561—2 (1917). Lockett and Shaw. Apparatus for purifying sewage etc. (156,816-7.) Jan. 26.  
22,774 (1919). Kowalski. See XVII.  
3496 (1920). Barron. Sand-filters. (156,424.) Jan. 19.

11,664 (1920). Fairweather (Lysekils Mekaniska Verkstads Akt.). Distilling water. (156,450.) Jan. 19.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

## APPLICATIONS.

Bosshard, Strauss, and Elektrochem. Werke. Production of condensation products. 1627—8. Jan. 10. (Ger., 9,8,18, 4,7,19.)  
Bräunig and Wohl. Production of glyoxal. 1241. Jan. 10. (Ger., 29,6,16.)  
Farbw. vorm. Meister, Lucius, u. Brünig. Manufacture of complex aurothiophenols. 1579. Jan. 10. (Ger., 22,4,16.)  
Farbw. vorm. Meister, Lucius u. Brünig. Manufacture of phlorobutyrophenone, phloroisobutyrophenone, and their homologues. 1580. Jan. 10. (Ger., 3,11,17.)  
Haakh. Manufacture of oxyarylaldehydes. 1576. Jan. 10. (Ger., 6,2,18.)  
Mauthner and Pfeifer. 1146. See III.  
Morgan and Vining. Production of aromatic aldehydes. 2480. Jan. 19.

## COMPLETE SPECIFICATIONS ACCEPTED.

2366 (1918). Steele and Robertson. See III.  
24,358 (1919). British and Foreign Chemical Producers (Rhein. Kampfer-Fabr.). Manufacture of *p*-cymene. (156,329.) Jan. 19.  
14,767 (1920). Inray (Monsanto Chem. Works). See IV.  
17,795 (1920). Boehringer Sohn. Obtaining the active ingredient of *Lobelia inflata*. (145,621.) Jan. 26.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## APPLICATIONS.

Dorian. Colour photography. 2720. Jan. 20. (Fr., 21,1,20.)  
Gorsky. Three-colour photography. 1879. Jan. 12.  
Kühn. Photographic printing processes. 1527. Jan. 10. (Ger., 20,7,16.)

## COMPLETE SPECIFICATION ACCEPTED.

9895 (1920). Dourlen and Chretien. Colour photography. (141,368.) Jan. 19.

## XXII.—EXPLOSIVES; MATCHES.

## APPLICATION.

Coleman. Method of making slow-burning matches. 1977. Jan. 13.

## COMPLETE SPECIFICATION ACCEPTED.

4597 (1920). Eschbach. Manufacture of initial explosive priming compositions. (156,429.) Jan. 19.

## I.—GENERAL; PLANT; MACHINERY.

*Silica gel; Adsorption [of vapours]* by — E. B. Miller. *Cham. and Mat. Eng.*, 1920, 23, 1155—1158, 1219—1222, 1251—1254.

THE phenomena of adsorption of vapours have been investigated with silica gel, prepared by coagulation of a colloidal solution of silicic acid, this material being specially applicable inasmuch as it is easily reproducible, is inert, and is stable even at high temperatures. The factors which influence the adsorption of vapours are the ratio of the partial pressure of the vapour to its vapour pressure at the temperature of adsorption, this ratio being termed the "corresponding pressure," and the compressibility of the adsorbed liquid, and to secure activity of the adsorbent it must have as large an internal volume as possible made up of spaces of minimum dimensions. Laboratory-scale experiments were carried out on the adsorption and recovery of sulphur dioxide, ether, acetone, benzene, water, and petroleum ether. In the case of sulphur dioxide the rate of flow was 40 c.c. per min. per g. of gel, giving a time of contact of 0.8 sec. For a certain period adsorption reached 100%, after which it gradually fell to zero; at the "break point" for all concentrations of the vapour the gel showed 57% saturation. Size of the gel particles affects adsorption. Recovery of the sulphur dioxide was effected by heating the gel to 100° C. and introducing air; in this way any desired concentration may be obtained. In experiments with volatile solvents these were recovered after adsorption by heating the gel to 105° C. and then admitting steam. The amounts recovered with ether, acetone, benzene, and petroleum ether were 84.7 and 90.4%; 82 and 93.5%; 86.5 and 95.5%; and 84.7 and 94.5%, respectively; the results are shown graphically. The adsorption of water by the gel is increased if the water content of the latter is 5—7%; to remove the water the gel is heated to 125° C. in a current of air. A plant for affecting adsorption on a semi-commercial scale is described. The results obtained with it prove the applicability of adsorption by silica gel to industrial processes such as air drying, recovery of gasoline from gas wells and prevention of loss in storage tanks, solvent recovery, recovery of vapours such as sulphur dioxide and nitrogen oxides, and separation of gases from mixtures.—W. J. W.

## PATENTS.

*Centrifuges.* C. A. Fesca & Sohn, Assees. of L. von May. E.P. (A) 137,827 and (a) 145,397, 12.1.20. Conv., 26.6.15 and 18.6.19.

(A) THE basket of a centrifugal machine is made without a top rim, and is mounted so that it can be raised against an annular disc which rotates in separate bearings and retains the solid matter. When the basket is lowered (without stopping the rotation) the solid material is automatically discharged. (B) The annular disc has a cylindrical upward extension forming a feed duct, above which is a fixed feed hopper with inlet and outlet valves controlled by one face of a double cam, the other face of which operates on the footstep bearing of the basket, so that the various motions are operated in correct sequence.—B. M. V.

*Acid-resisting receptacles, such as boilers, pipes, and the like.* F. Krupp A.-G. E.P. 145,732, 2.7.20. Conv., 17.6.18.

THE inner wall of the vessel is composed of acid-resisting iron, the outer wall of, e.g., ordinary iron, and the space between is filled with lead by casting.—B. M. V.

*Drying apparatus.* T. Allsop and W. W. Sibson, Assrs. to The Philadelphia Drying Machinery Co. U.S.P. 1,355,117, 12.10.20. Appl., 4.8.19.

A NUMBER of carriers for the goods to be dried are disposed within an enclosure so that their vertical ends form a number of transverse channels. Means are provided for circulating air in a number of independent courses each comprising adjacent channels.—J. S. G. T.

*Drying machine.* M. F. Mangelsdorff. U.S.P. 1,355,238, 12.10.18. Appl., 14.9.18.

HEATED air is admitted to a casing containing a series of superposed trays, which are perforated and divided into sections or leaves, each leaf being capable of rotation about an axis which is at right angles to the axes of the leaves of the tray next beneath. The sets of leaves are, at intervals, turned through 180° in succession, beginning at the bottom, the material being fed in a uniform layer to the top tray, and when dry, removed from the bottom of the casing by means that prevent escape of heated air.—B. M. V.

*Desiccating liquids; Process and apparatus for* — R. F. Barker. U.S.P. 1,362,590, 21.12.20. Appl., 23.6.19.

THE liquid is atomised and evaporated to a viscous state in a drying atmosphere, and collected on a moving heated surface, from which it is removed when completely dry.—B. M. V.

*Volatile solvents; Process of recovering* — U. J. Lebourveau and A. M. Taylor, Assrs. to Atlas Powder Co. U.S.P. 1,355,401, 12.10.20. Appl., 5.3.20.

THE solvent vapour is extracted from its mixture with air by means of acetone oil or aniline, which is then distilled and the solvent vapour condensed.—W. F. F.

*Air separator.* G. S. Emerick, Assr. to Emerick Mill Co. U.S.P. 1,355,596, 12.10.20. Appl., 7.1.19.

THE apparatus comprises a casing within which is a chamber containing an upper and a lower receptacle with a space between the two. Walls connected with and extending from the receptacles form an air chamber around the space between the receptacles. The material under treatment is fed into the upper receptacle, and air is supplied through an adjustable passage to the air chamber, and passes through the space between the receptacles and upwards through and around the upper receptacle, carrying with it the lighter particles which are deposited in and removed from the outer casing. The heavier particles pass downwards into the lower receptacle and are discharged through a separate outlet.

*Compound still.* A. C. Badger. U.S.P. 1,362,666, 21.12.20. Appl., 29.1.17.

A CONTAINER is provided with a heater at the upper part, a catch pan below the heater, and a condenser in the lower part. Outlets are provided from the catch pan and condenser, there is an inlet for liquid in the lower part of the container and an inlet pipe within the container leading to the condenser from the vapour space above the level of the liquid.—B. M. V.

*Fractionating-column.* L. V. Rogers. U.S.P. 1,363,435, 28.12.20. Appl., 7.7.19.

A FRACTIONATING column detachable from the boiling vessel is fitted with an inverted conical baffle over the vapour inlet at the bottom and with a number of horizontal perforated baffle discs.

—L. A. C.



*Gravity-kiln.* T. F. Anderson. U.S.P. 1,363,215, 28.12.20. Appl., 26.5.20.

A TUBULAR oven is arranged in a slanting position, so that the material therein will travel downwards by gravity, and is heated by hot gases in a channel which first surrounds the oven and then leads vertically away from it, so as to leave the top and mouth of the oven unobstructed.—B. M. V.

*Evaporator.* R. D. Keboe. U.S.P. 1,363,323, 28.12.20. Appl., 13.8.19.

In an evaporator the steam for heating is led vertically downwards through the top of the vessel to a heater in the lower part, and the entrainment separator below the vapour outlet at the top is arranged round the steam inlet so that the vapour must pass close thereto.—B. M. V.

*Mixing apparatus.* F. A. Browne, Assr. to The Barber Asphalt Paving Co. U.S.P. 1,363,561, 28.12.20. Appl., 12.4.17.

A ROTATING cylindrical drum is provided with sets of baffles on its interior surface, the baffles of any one set being substantially parallel to each other, but extending across the path of material coming from an adjacent set.—B. M. V.

*Gases, vapours and liquids; Apparatus for removing mechanically mixed solids from —.* R. Wussow and E. Schierholz. G.P. 324,260, 18.10.18.

THE liquid or gas is directed tangentially into a cylindrical apparatus provided with spiral guides. Outlet passages are connected with these guides round the circumference of the apparatus and are directed downwards. The particles of solid are driven against the periphery of the apparatus and pass into the outlet passages, while the purified liquids or gases pass down through the centre and out through a central outlet tube.—A. R. P.

*Pulveriser; Rotary —.* Soc. Milanese Impianti Industriali Cortese - Crespi - Squassi. E.P. 136,831, 17.12.19. Conv., 17.12.18.

SEE U.S.P. 1,339,499 of 1920; J., 1920, 476 A.

*Drying or partially eliminating moisture from materials; Method of and apparatus for —.* H. B. Lowden. E.P. 155,493, 11.3.20.

SEE U.S.P. 1,339,771 of 1920; J., 1920, 476 A.

*Drying-oven; Vacuum —.* J. D. Taylor. U.S.P. 1,365,207, 11.1.21. Appl., 31.3.20.

SEE E.P. 136,703 of 1919; J., 1920, 144 A.

*Lining for tanks.* F. Schuler. U.S.P. 1,355,360, 12.10.20. Appl., 10.6.19.

SEE G.P. 318,033 of 1918; J., 1920, 356 A.

*Separation of suspended matter from gaseous fluids; Electric means for —.* E. Möller, Assr. to The Chemical Foundation, Inc. U.S.P. 1,362,128, 14.12.20. Appl., 7.3.12.

SEE F.P. 449,337 of 1912; J., 1913, 495.

*Mixing liquid, semi-liquid, or viscid materials; Machine for —.* A. Sonsthagen. U.S.P. 1,363,368, 28.12.20. Appl., 4.3.20.

SEE E.P. 134,451 of 1919; J., 1920, 2 A.

*Feeding material through rotating cylinders while subjected to roasting, mixing, or similar operations; Means for —.* A. Sonsthagen. U.S.P. 1,363,369, 28.12.20. Appl., 4.3.20.

SEE E.P. 140,880 of 1919; J., 1920, 436 A.

*Furnaces; Apparatus for delivery of gas and air to —.* G. and J. Keith. U.S.P. 1,363,513, 28.12.20. Appl., 14.10.18.

SEE E.P. 120,076 of 1917; J., 1918, 756 A.

*Centrifugal separator.* K. and A. Ward. U.S.P. 1,363,699, 28.12.20. Appl., 11.6.18.

SEE E.P. 119,642 of 1918; J., 1918, 723 A.

*Levitation of granular and pulverulent material.* G. Gröndal. U.S.P. 1,363,970, 28.12.20. Appl., 15.7.19.

SEE E.P. 137,930 of 1919; J., 1920, 239 A.

*Drying liquids.* E.P. 155,927. See XIXA.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Sulphur in the coal bed; Distribution of the forms of —.* H. F. Yancey and T. Fraser. J. Ind. Eng. Chem., 1921, 13, 35—37.

ANALYSES of samples of coal from different parts of the coal face showed that pyritic sulphur is extremely irregularly distributed, but the organic sulphur is much more uniform. There appears to be no definite relationship between the occurrence of organic and of pyritic sulphur. High-sulphur coals are ordinarily higher in both types than low-sulphur coals, though organic sulphur makes up a greater percentage of the total sulphur in the case of low-sulphur coals. No difference was found in the percentage of organic sulphur in washed and unwashed coal, and no evidence was obtained of a concentration of organic sulphur in the coal immediately adjacent to pyrites deposits.—W. P.

*Sulphur content of coke and gas in the carbonisation of coal; Factors affecting the —.* A. R. Powell. J. Ind. Eng. Chem., 1921, 13, 33—35.

A DETAILED study has been made of the changes the pyritic and organic sulphur in coal undergo during carbonisation. Pyrites itself is completely decomposed at 1000° C., giving ferrous sulphide and free sulphur and a trace of pyrrhotite or magnetic iron sulphide. In coal the decomposition of the pyrites begins at 300° and is complete at 600°, the reaction reaching its maximum at 400°—500° C. From one-quarter to one-third of the organic sulphur is decomposed below 500° C., forming hydrogen sulphide. A small part of the organic sulphur gives volatile organic sulphur compounds which find their way into the tar. The remaining organic sulphur undergoes a decided change in character at 400°—500° C., and shows none of the properties of the original coal sulphur. Coke practically ceases to give off hydrogen sulphide above 600° C., but if hydrogen or gas containing hydrogen is passed through coke above 600° C., a further appreciable evolution of hydrogen sulphide is obtained. The reaction appears to be reversible, and for the removal of appreciable quantities of sulphur large quantities of the gas containing hydrogen must be passed through the coke at a rapid rate.—W. P.

*Colloidal fuels, their preparation and properties.* S. E. Sheppard. J. Ind. Eng. Chem., 1921, 13, 37—47. (Cf. J., 1920, 395 A, 413 A.)

TWO types of colloidal fuels are distinguished. The A-type is stabilised by means of a "fixateur" or protective colloid. Soaps, particularly of the alkaline-earth metals, such as calcium soaps, are used as fixateurs. The B-type is stabilised by "peptisation" (or partial solution) with tar or tar distillates. The B-type are more easily prepared than the A-type, but are not completely stabilised. Colloidal fuel prepared by peptisation will remain homogeneous for 3 to 4 weeks, after which the oil and tar begin to separate. A second method of peptisation consists in partial oxidation of the cellulosic constituents of the coal. The machinery

for compounding these fuels consists of a mill for pulverising the coal, coke, etc., storage and blending tanks for the oil bases, and mixing vessels. The cost of manufacture is reckoned approximately at \$1.50 per ton, inclusive of fixateur.—W. P.

*By-product coke, anthracite, and Pittsburgh coal as fuel for heating houses.* H. Kreisinger. J. Ind. Eng. Chem., 1921, 13, 31–33.

THE comparative values of by-product coke, anthracite, and Pittsburgh coal were examined, two steam boilers of the size ordinarily used for heating an average 7-room house being used for the tests. The efficiency obtained with the coke was a little better than that obtained with anthracite and 10–17% better than that obtained with Pittsburgh coal. Per lb. of fuel burned the coke was about 15% better than the Pittsburgh coal, and the anthracite about 9% better than the coke.—W. P.

*Low-temperature coking of Utah coals.* O. Monnett. Chem. and Met. Eng., 1920, 23, 1246–1249.

FIFTEEN 30-lb. samples of different varieties of coal were analysed and examined in regard to their coking power in a 20-g. by-product apparatus at 540° and 700° C., the best ten being further tested in an apparatus of 8-lb. capacity at a maximum temperature of 1000° C. The tabulated results give the amounts of tar, coke, and gas, as well as the composition of the last, and the calorific power of the coal, and analyses of the coal are also quoted. The conclusion arrived at is that Castle Grade coal gives a fair grade of coke, but Sunnyside is the best coking coal, and a good mixture consists of equal parts of Standard and Sunnyside.—W. J. W.

*Canadian lignite; Carbonisation of —.* E. Stansfield. J. Ind. Eng. Chem., 1921, 13, 17–23. (Cf. J., 1919, 67 A, 491 A.)

NEW plant in the course of construction is described. This consists essentially of a strongly heated retort floor, inclined at an angle slightly steeper than the angle of repose of the crushed lignite. The material flows down the heated surface from a hopper at the top, passing under a succession of haffle plates, which control the thickness of the layer. The rate of flow of the material is controlled entirely by the rate of withdrawal from the bottom of the retort. The retort floor should be hottest at the bottom of the retort. Satisfactory results have been obtained with a small model of the same design capable of treating 200 lb. of raw lignite per hr.—W. P.

*Ammonia; Influence of steam and gases on the yield of — from the carbonisation of coal and oil shales.* A. J. Franks. Chem. and Met. Eng., 1920, 23, 1149–1154.

IN the carbonisation of coal at low temperatures (not exceeding 500° C.), the predominance of hydrogen over ammonia, and inert gases present, operate against dissociation of the ammonia, and if steam is introduced into the process it exerts a mechanical action in removing the ammonia from the reaction zone before decomposition takes place. At high temperatures the protective action of hydrogen is still more evidenced. At high temperatures with steam, apart from the normal effect of the hydrogen, dissociation of the steam gives rise to formation of large quantities of nascent hydrogen, which effect the synthetic production of ammonia, as indicated by Tervet (J., 1933, 445).—W. J. W.

*Producer fire; Effect of cooling the — on gas production.* H. Koschmieder. Brennstoff-Chem., 1921, 2, 3–5.

THE heat balance on steaming the producer fire is examined. With large quantities of steam there is

an increase in gas yield, but a decrease in calorific value. At the same time there is an increase in the yield of ammonia.—W. P.

*Acetylene; Pyrogenic decomposition of — in contact with metallic catalysts.* E. Tiede and W. Jenisch. Brennstoff-Chem., 1920, 2, 5–8.

THE temperature at which decomposition of acetylene commenced in contact with different metallic catalysts was determined, and also the percentage decomposition with each metal at 800° C. The most active metals in promoting decomposition were iron, cobalt, nickel, copper, and manganese. The differences observed in the action of different metals are attributed to their varying solvent power for hydrogen, except in the case of the alkali metals, the activity of which is due to the ease with which they form carbides. (Cf. J.C.S., Feb.)—W. P.

*Coal gas and air; Internal energy of inflammable mixtures of — after explosion.* W. T. David. Proc. Roy. Soc., 1920, A, 98, 303–318.

ABOUT 10% of the heat of combustion of coal gas in mixtures with air, has not been converted into thermal energy when the maximum pressure of the explosion has been reached, and an after-burning continues for at least 0.25 sec. after this maximum pressure has been attained. The following figures show the distribution of the energy of combustion at the moment of maximum temperature for mixtures of gas and air containing respectively 15, 12.4, and 9.7% of coal gas:—Internal thermal energy, 81, 78, 72.5% of the total heat of combustion of the coal gas; available chemical energy in unburnt coal gas, 10, 12, 9.5%; heat loss to walls of vessel, 9, 10, 18%. (Cf. J.C.S., Feb.)—J. F. S.

*Combustions; Mechanism of some —.* H. von Wartenberg and B. Sieg. Ber., 1920, 53, 2192–2202.

CARBON monoxide must be moist before it will react with oxygen at ordinary flame temperatures, but the amount of water necessary to promote explosion of the ideal mixture is just the same whether the oxygen is provided as such or as nitrous oxide, and is roughly that quantity which corresponds with a partial pressure of 0.5 mm. The carbon monoxide flame contains hydrogen as well as formic acid (Wieland, J., 1912, 333 A), and therefore Dixon's theory of the mechanism of the combustion (J., 1880, 370), in which the first stage is the union of carbon monoxide and water to formic acid, is completely proved. The union of hydrogen and oxygen at 600°–1000° C. also begins by a direct addition, namely the formation of hydrogen peroxide, for the amount of this which may be proved to be present in the products when the combustion tube is rapidly cooled, is about one million times as great as would be formed by the secondary oxidation of water under the same conditions. The hydrogen peroxide, however, rapidly decomposes into water and oxygen, some of which changes to ozone, which persists better than the hydrogen peroxide. (Cf. J.C.S., Feb.)—J. C. W.

*Gasoline losses due to incomplete combustion in motor vehicles.* A. C. Fieldner, A. A. Strauh, and G. W. Jones. J. Ind. Eng. Chem., 1921, 13, 51–58.

ANALYSES of exhaust gases have been carried out in order to examine the gasoline losses in motor engines due to imperfect combustion. The composition of the exhaust gases from individual machines varies greatly, the controlling factor being the air-gasoline ratio supplied to the cylinders. The percentage of carbon monoxide in the exhaust gas lies between 5 and 9%, the average for 23 cars tested being 6.7%, and the heat lost in unburnt

gases passing into the exhaust is about 30% of the total heat value of the gasoline supplied. The maximum loss takes place when operating for full power on heavy load with rich mixtures.—W. P.

*Oils and other liquids; Viscosity of — as a function of temperature.* H. Schwedhelm. Chem.-Zeit., 1921, 45, 41–42.

DETERMINATIONS of the viscosity of mineral oils at different temperatures showed that the viscosities may be calculated approximately by the formula  $\eta/\eta' = (z/z')^{H/t}$ , where  $z$  and  $z'$  are the absolute viscosities at temperatures  $t$  and  $t'$ , and  $G$  and  $H$  constants.  $G$  and  $H$  have the following values:—mineral lubricating oils, 0.01887–0.02357, 1.01435–1.01858; rape oil, 0.01887, 1.01218; water, 0.00145, 1.01278; alcohol, 0.000160, 1.00441.—W. P. S.

*Coke-oven refractories.* Hancock. See VIII.

#### PATENTS.

*Furnaces; Pulverised fuel —.* L. H. Bergman. E.P. 155,731, 17.3.20.

AIR is injected into a combustion chamber through two concentric nozzles, and powdered fuel is also injected into the central nozzle to mix with the air. The mixed streams of air and fuel are directed downwards towards the forward end of the combustion chamber, and are then diverted to the rear of the chamber and thence through an outlet leading to a boiler.—W. F. F.

*Furnace and the process of combustion of pulverulent and other fuel adapted for steam boilers.* F. Seymour. U.S.P. 1,355,172, 12.10.20. Appl., 6.1.19.

FUEL and air are mixed in the exact proportions that are necessary for complete combustion and introduced as a blast into the combustion chamber, which is provided with walls of refractory material. The walls are heated to incandescence and the combustible material is ignited in contact with them. The maximum combustion temperature is obtained, and the gas is brought into contact with a steam generator after combustion is substantially complete. The melted residue is discharged through a water-cooled opening into a water-cooled ash-pit without agglomeration.—W. F. F.

*Distillation or gasification of organic matter or minerals containing organic matter; Process of and oven for the continuous —.* F. Rippl. Assr. to The Chemical Foundation, Inc. U.S.P. 1,355,268, 12.10.20. Appl., 8.8.16.

THE material is placed in receptacles and moved along an oven passage which is heated externally. In the preheating zone the oven is heated in one part only at its sole and in another part only at its lateral walls, whilst in the distillation zone it is heated at its sole as well as its lateral walls. In the final zone the material is cooled by means of the combustion air which is supplied to the oven.—W. F. F.

*Gas producers.* J. M. Wallwin. E.P. 155,679, 14.10.19.

In a suction gas producer having a circular stepped grate with central apertures in the grate plates, the plates are in the form of concentric truncated cones to facilitate the passage of ash. The bottom plate includes a movable piece to allow of clinking.—C. I.

*Gas producer.* Bergmann Elektrizitäts-Werke A.-G. G.P. 323,769, 10.11.17.

THE fire-grate of a gas producer is roof-shaped and is movable along the direction of the ridge. In order to prevent conglomeration and piling up of

the coke, and to effect its reduction to small fragments, the fire-grate is further provided with projections which operate against other fixed projections and thereby impart an active movement to the mass.—W. J. W.

*Hydrocarbons; Apparatus for treating —. Art of cracking [petroleum] hydrocarbons.* J. W. Coast, jun., Assr. to The Process Co. U.S.P. (A) 1,355,311 and (B) 1,355,312, 12.10.20. Appl., 5.5 and 27.9.17.

(A) THE vapour from a still bubbles through a body of hydrocarbon oil contained in a reservoir and then through a reflux condenser which returns the condensed liquid to the reservoir, whence it flows by gravity through an overflow pipe into the still. The vapour not condensed in the reflux condenser passes into a main condenser. (B) In the apparatus described in U.S.P. 1,353,316 (J., 1920, 742 A), the oil is filtered both before and after passage through the pipe element to remove coke-forming matter.—L. A. C.

*Gasoline; Manufacture of — [from oil well gas].* G. H. Taber, jun., Assr. to Mexican Sinclair Petroleum Corp. U.S.P. 1,363,487, 28.12.20. Appl., 24.5.19.

OIL and gas are withdrawn from an oil well under high pressure; a portion of the gas is released under a lower pressure sufficiently high to prevent much volatilisation of gasoline, a further portion is then released at a still lower pressure, and gasoline is extracted from this portion.—L. A. C.

*[Motor] fuel; Method of forming a liquid —.* A. Hayes, Assr. to U.S. Industrial Alcohol Co. U.S.P. 1,363,870, 28.12.20. Appl., 18.6.19.

ALCOHOL vapour is passed into a mixture of about 50 pts. of kerosene, 14 pts. of benzol, and 3 pts. of ether.—L. A. C.

*Coking retort ovens.* W. J. Mellersh-Jackson. From The Koppers Co. E.P. 155,316, 23.6.19.

SEE U.S.P. 1,312,372 of 1919; J., 1919, 710 A.

*Lubricating oil; Process of purifying —.* C. H. Hapgood, Assr. to The De Laval Separator Co. U.S.P. 1,363,784, 28.12.20. Appl., 23.4.19. Renewed 17.11.20.

SEE E.P. 142,089 of 1920; J., 1921, 38 A.

*Gas retorts and the like; Machines for charging vertical —.* Goodall, Clayton, and Co., Ltd., G. R. Payne, and J. A. Archdale. E.P. 156,324, 6.10.19.

*Furnaces [gas producers]; Rotary grates for —.* J. Lambot. E.P. 141,056, 31.3.20. Conv., 3.4.19.

*Hydrogen.* U.S.P. 1,363,488. See VII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### PATENTS.

*Wood alcohol; Process for the manufacture of —.* L. F. Hawley. U.S.P. 1,363,730, 28.12.20. Appl., 19.10.20.

WOOD is treated with sodium carbonate and subjected to destructive distillation.—L. A. C.

*Retorts; Vertical — [for distillation of bituminous materials].* K. Barthel. G.P. (A) 323,958 and (B) 323,959, 26.9.18.

(A) THE material is delivered from a preheating chamber, by means of a revolving hollow drum, to the uppermost of a series of superposed plates in a

vertical retort, and is moved over the plates and from one plate to the next lower one by rotating scrapers. In the production of low-temperature tar from bituminous materials, the hollow shaft carrying the scrapers serves as a superheater for the steam which is introduced, and a better distillation is thereby secured. At the same time the temperature of the gases is appreciably lowered and their decomposition prevented. (8) In a vertical retort provided with plates and scrapers, the products of distillation from different zones are drawn off separately and their fractionation is thus facilitated.—W. J. W.

*Incandescence electric lamps; Filament for* —. R. E. Myers and R. D. Hall, Assrs. to Westinghouse Lamp Co. U.S.P. 1,363,162, 21.12.20. Appl., 14.5.15.

A FILAMENT which tends to offset in use consists of tungsten and at least two of a group of substances including the oxides of the alkalis, alkaline earths, beryllium oxide, calcium oxide, and titanium oxide. —J. S. G. T.

*Distilling coal and other materials; Process of and apparatus for* —. W. Thomas. U.S.P. 1,365,123, 11.1.21. Appl., 15.11.16. Renewed 28.10.20. SEE E.P. 110,217 of 1916; J., 1917, 1230.

### III.—TAR AND TAR PRODUCTS.

*Sulphur in coal tar oils.* Weissgerber. Brennstoff-Chem., 1921, 2, 1—3.

THE sulphur in coal tar oil exists mainly as compounds containing the thiophene ring, either alone or in the form of condensation products. These compounds are very stable and so are particularly difficult to remove. Metals have been used for the purification of the oils. The alkali metals act vigorously at comparatively low temperatures (110°—120° C.), but the reaction is much slower with the heavy metals, and nickel steel and cast iron are practically inactive. The use of sodium is out of the question, not only on account of its prohibitive price, but also because of the presence of phenols in the oils. For practical purposes copper and zinc have been much used, but here also the cost of purification is high.—W. P.

*Phenols from low-temperature tar; Action of* — upon metals. U. Ehrhardt and G. Pfeiderer. Brennstoff-Chem., 1921, 2, 9.

THE solubility of metals in the oils from low-temperature tar was determined. The oils used were obtained by mixing 100 c.c. of first runnings, 600 c.c. of the fraction of b.p. 150°—250° and 300 c.c. of b.p. 250°—270° C. The mixture was saturated with water and was shaken with the metal to be tested (in most cases in the form of strips) in contact with air for a period of 4 weeks. The amounts dissolved in mg. per sq. cm. were as follows: copper, 6.5; brass, 0.6; German silver, 0.9; zinc, 1.2; galvanised iron, 0.73; nickel (wire), 0.09; nickel-plated brass, 0.17; lead, 1.1; aluminium, 0.0; tinplate, 0.22; sheet iron, 0.51; pickled sheet iron, 0.04; silicon iron (18%), 0.0; V2A-steel (Krupp), 0.0.—W. P.

*Sulphonic acids; Identification of* — in the form of salts formed with aromatic bases. C. F. van Duin. Rec. Trav. Chim., 1921, 40, 99—102.

SULPHONIC acids can be identified by means of their salts with aromatic bases (cf. Ambler and Wherry, J., 1920, 815 A) and these salts can be analysed by titration with N/10 sodium hydroxide in the presence of phenolphthalein as indicator. This has been applied successfully in a number of cases. (Cf. J.C.S., Feb.)—W. G.

### IV.—COLOURING MATTERS AND DYES.

#### PATENTS.

*Dyestuffs; Process of producing* —. E. Hart and I. J. Stewart. E.P. 155,726, 21.2.20.

FINELY divided vegetable matter, such as the wood, bark, leaves, or the like of certain trees and shrubs, is heated with a solution prepared by boiling sodium carbonate and lime in water and separating the calcium carbonate by filtration. When extraction of the colour is complete, the solution is filtered and may be used direct for dyeing or may be concentrated to a paste or to dryness. A wide range of dyestuffs can thus be extracted from different varieties of the following species: eucalyptus, eucarpus, acacia, mallotus, casnarina, banksia, and aster.—L. A. C.

*Disazo dye.* T. H. Leaming and T. M. Susemihl, Assrs. to National Aniline and Chemical Co. U.S.P. 1,363,386, 28.12.20. Appl., 1.7.19.

CLAIM is made to a disazo dye having the formula,  $(4)NH_2.C_6H_4.N:N.C_6H_4(1-OH,8-NH_2,5-SO_3H)(7)N:N.C_6H_4(4)NH_2$ , which yields p-phenylenediamine and triamino-3-naphthol-4-sulphonic acid on reduction with stannous chloride and hydrochloric acid, and dyes cotton, wool, and natural or artificial silk, greenish shades of black which can be developed on the fibre. —L. A. C.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*"Ashi" fibre; Microscopical examination, chemical composition, and reactions of* —. M. Ishikawa. Kōgyō Kwagaku Zasshi (J. Chem. Ind. Tokyo), 1920, 23, 1153—1157.

"ASHI" reed fibre resembles straw in regard to the kind of cells, chemical composition, and reactions. It gave the following analytical results: Moisture 11.2%, water-soluble matter 3.78%, ash 1.24%, fat and wax 0.96%, lignin 14.7%, cellulose 49.8%, and pectin etc. 18.32%.—K. K.

*Mechanical wood pulp; Determination of* —. M. G. Kotibhasker. J. Soc. Dyers and Col., 1921, 37, 11—13.

THE absorption of p-nitraniline by mechanical wood pulp is constant under defined conditions, experiment indicating that pure mechanical wood pulp in thin slices treated with the reagent in the proportion of 2 g. to 40—60 c.c. of an approximately 0.1% solution in dilute hydrochloric acid shows in 6—24 hrs. a constant absorption of 0.56—0.57%. The excess of p-nitraniline is determined by adding a known excess of titanous chloride to an aliquot portion of the solution, say 10 c.c., boiling to effect reduction, and titrating back the excess of titanous salt with standard solution of iron alum. By this means the mechanical wood in an unknown sample can be determined with an error of not more than about 2%. The absorption of p-nitraniline by jute also shows a constant figure of 0.35—0.37%. —G. F. M.

*Sulphite-cellulose waste liquors; Extraction of* — with ether and benzene. B. Holmberg. Svensk Kem. Tidskr., 1920, 32, 56—67. Chem. Zentr., 1920, 91, IV., 753.

THE extraction of sulphite liquors with ether yields a tarry substance and a white crystalline compound, termed sulphite liquor lactone,  $(C_6H_5O_2.OCH_3)_2$ . The latter forms square plates or flat prisms, m.p. 250°—255° C.,  $[\alpha]_D^{20} = -192^\circ$  (in acetone at room temperature), soluble in ether, alcohol, and methyl

alcohol, very soluble in acetone, but only slightly soluble in water. Sulphuric acid converts the lactone into a monosulphonic acid; on subsequent treatment with water a hydroxy-acid is formed. The lactone is only slowly dissolved by sodium carbonate solution, but is readily soluble in sodium or potassium hydroxide, from which it is precipitated by carbon dioxide. The hydroxy-acid has the formula  $C_7H_{12}(OH)(OCH_2)CO_2H$ ;  $[\alpha]_D = +280^\circ$  (in acetone at normal temperature). The lactone combines readily with diazonium salts in alkaline solution. It is probably the lactone of diguaiacoltetramethylcarbinolcarboxylic acid.

—W. J. W.

*Sericin.* Türk. See XIXA.

*Adsorption by cellulose.* Koltboff. See XXIII.

#### PATENTS.

*Textile fibres; Process of producing* —. R. A. Marr. U.S.P. 1,362,723, 21.12.20. Appl., 26.3.17. Renewed 10.5.20.

PLANT structures containing fibrous material are digested with a solution of a zinc salt, the digestion being stopped before the binding substances which hold the fibres together as filaments are attacked.

—A. J. H.

*Woody constituents of vegetable fibres; Process for removing* — by carbonising. Deutsche Wollentfettung A.-G. G.P. 325,885, 1.3.19.

THE material is immersed in dilute acid, after which it is rinsed with cold water before drying by heat. Bast fibres absorb the acid more readily than woody constituents, but also give up the absorbed acid more easily on washing. For the treatment of flax and hemp comings and waste derived from the spinning of bast fibres, sulphuric acid of 1° B. (sp. gr. 1.007) is effective.—W. J. W.

*Cellulose solutions; Process for preparing* —. Z. Ostenberg. U.S.P. 1,355,415, 12.10.20. Appl., 1.11.16.

CELLULOSE is dissolved in a mixture of calcium chloride with sulphuric acid of more than 60% strength.—A. J. H.

*Cellulose or the like; System for the treatment of* —. R. A. Kocher. U.S.P. 1,362,875, 21.12.20. Appl., 27.2.17.

THE apparatus consists of a container, into which halogen acid gas may be forced under pressure, and provided with means for agitating and varying the temperature of the material therein. Means are also provided for removing the halogen acid gas and halogen acid solution from the container, for separating these and for returning the halogen acid solution, independently of the halogen acid gas, for the treatment of the material.—A. J. H.

*Acetate of cellulose; Manufacture of solutions, compositions, or preparations having a basis of* —. H. Dreyfus. U.S.P. 1,363,763, 23.12.20. Appl., 7.7.19.

SEE E.P. 131,669 of 1918; J., 1919, 834 A.

*Semi-cellulose; Method of manufacture of* —. T. and E. Kittelsen. U.S.P. 1,365,039, 11.1.21. Appl., 5.4.19.

SEE G.P. 324,053 of 1919; J., 1920, 779 A.

*Paper-making machines.* Process Engineers, Inc., Assees. of W. E. Metcalf. E.P. 145,440, 18.6.20. Conv., 9.3.15.

*Paper pulp reclaimers.* C. S. Bird. E.P. 146,891, 6.7.20. Conv., 21.1.15.

*Removing fibrous material from the bottom of [pulp] digesters used in paper making.* W. M. Wallace. E.P. 156,025, 10.3.20.

*Cellulose.* G.P. 300,703. See XXII.

## VL.—BLEACHING; DYEING; PRINTING; FINISHING.

#### PATENTS.

*Dyeing raw fibrous stock; Method for* —. P. MacIntyre. U.S.P. 1,355,797, 12.10.20. Appl., 15.9.19.

THE raw material in bulk is treated with a dye liquor, and the dye is then oxidised by forcing an oxidising agent through the mass.—A. J. H.

*Washing and drying clothes; Process for* —. A. R. Willford, Assr. to The Electric Ozone Co. U.S.P. 1,362,658, 21.12.20. Appl., 8.3.17.

AT all stages in the washing process (washing, water extracting operation, mangling, and tumbling and shaking operations) the clothes are subjected to the action of ozone, and they are afterwards dried in a room containing ozonised air.

—A. J. H.

*Bleaching agent.* E.P. 147,535. See VII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Nitrites and nitrates; Reduction of* —. O. Baudisch and P. Mayer. Biochem.-Zeits., 1920, 107, 1—43.

NITRATES are reduced quantitatively by excess of ferrous hydroxide in neutral and alkaline solution. In boiling solutions made alkaline with carbonates, nitrous oxide and ammonia are chiefly formed. In boiling caustic alkali solution the nitrite is reduced quantitatively to ammonia. Alkali nitrates are reduced quantitatively to ammonia by ferrous hydroxide in neutral solution and in alkaline solution containing 28% of sodium hydroxide. Starting with neutrality the amount of nitrate reduced diminishes with addition of alkali until the alkali content is 6.5% NaOH when a minimum is reached; beyond that limit the amount reduced increases with the increase of alkali until the maximum is reached at 28% NaOH. Up to a concentration of 6.5% NaOH the presence of oxygen is necessary for the reduction with ferrous hydroxide. With higher alkali concentrations reduction also takes place in the absence of oxygen. Up to an alkali concentration of 6.5% NaOH the amount of nitrate reduced is proportional to the amount of oxygen present in solution or absorbed by the ferrous hydroxide. The above observations may be utilised for the estimation of nitrites and nitrates separately and when together.—S. S. Z.

*Salts; Double decompositions of* — and the phase rule. E. Rengade. Comptes rend., 1921, 172, 60—62. (Cf. J., 1917, 961.)

IN a discussion of Raveau's work (J., 1920, 819 A) the author considers the effect of the addition of a small amount of water to a mixture of sodium nitrate and ammonium chloride and shows that at the ordinary temperature there are only two ternary mixtures,  $NaNO_3-NH_4NO_3-NH_4Cl$  and  $NaNO_3-NH_4Cl-NaCl$ , which can exist without change in contact with a small amount of water. All other mixtures of the two, three, or four salts containing the four ions will decompose giving.

according to the conditions, one or other of the ternary mixtures mentioned.—W. G.

*Hydrogen cyanide; Detection of* — in air. A. Sieverts and A. Hermsdorf. Z. angew. Chem., 1921, 34, 3—5.

**FILTER-PAPER** is dipped in a solution of copper acetate and benzidine acetate and then exposed in the atmosphere to be tested; a blue colour appears on the paper within 7 secs., if the air contains more than 15 mg. of HCN per ch. m. The copper acetate solution (286 g. per l.) and benzidine acetate solution (475 c.c. of cold saturated benzidine acetate solution and 525 c.c. of water) are kept separately and equal volumes are mixed immediately before use.—W. P. S.

*Hydrogen arsenide; Preparation of* — of high purity, and its quantitative determination. H. Thoms and L. Hess. Ber. Deuts. Pharm. Ges., 1920, 30, 483—489.

Pure hydrogen arsenide is conveniently obtained by the action of water or acids on calcium arsenide, and the difficulty and danger of preparing this latter substance can be entirely obviated by adding a neutral diluent such as sand (5.5 kg.) to the mixture of powdered arsenic (3.1 kg.) and coarse calcium filings (2.4 kg.). The mixture is placed in a sheet iron container enclosed in a second vessel, and ignited by means of a mixture of potassium chlorate and magnesium. The mass glows intensely, but no flame and but little arsenious oxide is produced. The arsenide is removed when cold and ground to a coarse powder which may then be used instead of sand as diluent in the same proportion in succeeding preparations. The evaluation of calcium arsenide is carried out in a nitrometer charged with brine, the hydrogen arsenide being absorbed from the evolved gas by cupric chloride solution. Calcium arsenide prepared as above gives about 37—38% of hydrogen arsenide containing about 0.004% by weight or 14% by vol. of hydrogen. The remainder of the arsenic in the metallic arsenide appears as a brown powder (probably solid hydrogen arsenide) in the reaction vessel. Aqueous solutions of hydrogen arsenide undergo rapid decomposition with the formation of metallic arsenic which remains in colloidal solution. The decomposition can be followed by titrating the solution with N/100 iodine, when the arsenide is oxidised first to arsenious acid, and then, if the solution is rendered alkaline with a bicarbonate, to arsenic acid in the usual way. (Cf. J.C.S., Feb.)—G. F. M.

*Bromine; Technology of* —. W. Hüttner. Chem.-Zeit., 1921, 45, 49—51.

A DESCRIPTION of methods used for the manufacture of bromine; the general process consists in the decomposition of bromides by chlorine (cf. Kubi-erschky, J., 1914, 135), but the electrolytic process is used in a few works. In Germany, the production of bromine rose from 7500 kg. in 1867 to 866,000 kg. in 1911; during the same period, the production in America increased from 5000 to 3,236,000 kg.—W. P. S.

*Ammonia.* Franks. See IIA.

*Mercuric chloride and lead salts.* Sasse. See XXIII.

#### PATENTS.

*Bleaching and disinfecting agents; Production of* —. G. Kereszty and E. Wolf. E.P. 147,535, 3.3.20. Conv., 12.7.19.

A MIXTURE of 1 pt. of basic magnesium hypochlorite and 3 pts. of sodium bicarbonate, which is stable in the dry state, yields a solution which after 10 mins. contains 50%, and after 4 days 90% of the active chlorine of the hypochlorite. The slow decompo-

sition compensates for the loss of chlorine on standing, and the mixture thus gives a solution of which the chlorine content is maintained better than in the case of other bleaching agents.—C. I.

*Alkali-metal compounds; Process of producing water-soluble* — from water-insoluble substances. H. S. Blackmore. U.S.P. 1,355,381, 12.10.20. Appl., 19.8.17.

INSOLUBLE minerals containing alkali compounds are treated with a non-acid silicofluoride.—C. I.

*Alkali-metal compounds; Process of producing water-soluble* — from water-insoluble substances. H. S. Blackmore, Assr. to Kali Co. of America. U.S.P. (a) 1,355,588 and (b) 1,355,794, 12.10.20. Appl., 14.12.18.

(A) SILICATES containing potassium are exposed to the action of a silicofluoride of a metal of the iron group, whereby the potassium is converted into a compound soluble in hot water. (B) Alkali compounds are extracted from insoluble silicates by exposing them to the action of an alkali silicofluoride, separating the resulting soluble product and treating it with a chemical reagent to produce a still more soluble alkali compound.—A. R. P.

*Alkali-metal salt mixtures; Process of treating* —. J. A. Cullen. U.S.P. (a) 1,363,091 and (b) 1,363,092, 21.12.20. Appl., 26.3.19.

(A) IN potassium-salt mixtures containing carbonates, the latter are precipitated, and a soluble sulphate is added to the solution in excess of the amount required to combine with the potassium which is not already united to the sulphate radicle. (B) After precipitating the carbonates with lime, a soluble haloid salt is added to furnish the acid radicle for the potassium. In both cases the potassium salt is recovered by concentration and crystallisation.—W. J. W.

*Hydrocyanic acid; Process of recovering* —. G. H. Buchanan, Assr. to American Cyanamid Co. U.S.P. 1,355,884, 12.10.20. Appl., 12.11.19.

AN impure cyanide containing 40% of calcium chloride and 15% of lime is made into an emulsion with water, treated with a dilute mineral acid, and heated.—C. I.

*Salts; Process for decomposing* — in pans. F. Brandenburg. G.P. 324,259, 27.10.17.

IN the working of spent pickle liquor to ferric oxide, the decomposition vessel is charged with previously prepared ferric oxide, heated, and the spent liquor then added gradually and continuously with constant stirring. A similar process is applicable to the treatment of waste liquors containing ferrous sulphate, magnesium chloride, sodium bisulphate, etc.—A. R. P.

*Sulphur; Purification of* —. J. J. Hood. E.P. 155,692, 25.10 and 17.11.19.

A SOLUTION of sulphur in carbon bisulphide, such as that obtained from spent oxide, is purified by agitation with 1—2% by vol. of sulphuric acid (sp. gr. 1.8), whereby a considerable quantity of tarry matter is precipitated. The mixture is then, either before or after filtration, agitated with 5—10% by vol. of water and the aqueous layer containing the impurities removed. Finally, the sulphur solution is filtered through alumina or bauxite and the carbon bisulphide evaporated, leaving a residue of pure sulphur.—A. R. P.

*Hydrogen; Process for producing* —. R. H. Uhlinger. U.S.P. 1,363,488, 28.12.20. Appl., 20.4.20.

A HYDROCARBON heated to 500°—900° C. is intro-

duced into a decomposition chamber maintained at 1100°—1300° C.—L. A. C.

*Nitric acid; Production of concentrated nitrous gases and —.* Norsk Hydro-Elektrisk Kvaestofaktieselskab. E.P. 137,071, 24.12.19. Conv., 21.10.16.

SEE U.S.P. 1,316,950 of 1919; J., 1919, 817 A.

*Zinc oxide; Treating —.* New Jersey Zinc Co., Assees. of F. G. Breyer and E. H. Bunce. E.P. 138,924, 12.2.20. Conv., 9.8.18.

SEE U.S.P. 1,339,544 of 1920; J., 1920, 489 A.

*Electrical treatment of gases.* U.S.P. 1,363,827. See XI.

### VIII.—GLASS; CERAMICS.

*Silica glass; Double refraction and crystalline structure of —.* Lord Rayleigh. Proc. Roy. Soc., 1920, A, 98, 284—295.

GLASSES in general have no double refraction, except that due to bad annealing, but silica glass shows weak double refraction (about 1/60th that of crystalline quartz), which can only be accounted for by accepting that the glass has a crystalline structure. In a mass of silica which has been melted, but not drawn or blown, the structure consists of doubly refracting grains, with dimensions about 0.5 mm., oriented at random. The grains are very persistent and individually survive a re-melting of the material. If the grained material is drawn out while soft, the grains are elongated into crystalline fibres or ribbons. These fibres always give "straight extinction" in the polariscope, and their length is along one axis of the ellipsoid of optical elasticity, but apparently not always along the same axis. If the material is bent or twisted, the fibres follow its course unbroken, and are always extinguished in the polariscope if the nicols are set along the tangent and normal to their direction at any point. Fused silica sometimes contains isolated small inclusions of quartz, with angular outlines, which have escaped vitrification. These are conspicuous in the polariscope by the strain effects they produce in the surrounding glass.—J. F. S.

*Refractory materials used in coke-oven construction; Properties of —.* W. C. Hancock. Iron and Steel Inst., Carnegie Scholarship Mem., 1920, 41—63.

TEST-PIECES were made of Stourbridge clay mixed with varying proportions of "grog" made from the same clay fired to about 1400° C., sometimes with the addition of ganister to increase the silica content. An abrasion test was devised in which one test-piece was rigidly fixed whilst another was caused to pass backwards and forwards over its surface. When both surfaces had the same composition the abrasion was the same for both. Clay surfaces without grog showed least abrasion. Fine-grained surfaces suffered less abrasion than coarse-grained, whether the coarser grain was due to an increase in quantity or size of the grog. Increasing the size of the grog particles tended to decrease the porosity and increase the density. Tests on permeability to air at ordinary temperatures showed a decreasing permeability as the proportion of grog was increased, and also a decreasing permeability as the particles became finer. A few heat expansion tests were made with the following results: Clay, 0.33% at 750° C., 0.70% at 950° C., and 0.74% at 980° C.; mixture of 80% of clay and 20% of 30-mesh grog, 0.34% at 800° C. and 0.47% at 900° C.; mixture of 60% of clay and 40% of 1/4 in.-mesh grog, 0.40% at 860° C. and 0.46% at 990° C. The presence of soluble salts in coals has

a great influence on the life of the lining of coke ovens. By washing the coal with water a certain proportion of the salts can be removed, according to the size of the particles and the method of washing. Thus with three coals, ground to pass a 1/4 in.-mesh sieve, 49%, 48%, and 73% respectively of the soluble salts were removed by treatment with warm water on a hot plate for 8 hrs. No relation between the chlorine content and total salt content of different coals could be established. By heating fireclay with coal at 1450° C. the alkali content of the fireclay was considerably increased when unwashed coal was used, the increase being less with washed coal. The presence of moisture appeared to favour the incorporation of alkali into the refractory.—E. H. R.

*Clays; Effect of aluminium chloride upon —.* H. P. Reinecker and J. S. George. J. Amer. Ceram. Soc., 1920, 3, 994—996.

INCREASING amounts of aluminium chloride, varying from 0.05 to 0.5% of the dry weight of the clays, were added to North Carolina and Georgia kaolins and to Tennessee ball clay No. 5. In a subsequent series of experiments sufficient ammonia was added to precipitate the alumina. The mixtures were made into briquettes and bars, and the water of plasticity, drying shrinkage, and modulus of rupture in the dry state determined. Aluminium chloride, with or without ammonia, did not noticeably affect the plasticity, the greatest change being observed with 0.4 and 0.5% Al<sub>2</sub>Cl<sub>3</sub>+ammonia. The addition of aluminium chloride reduced the drying shrinkage of the two kaolins, but the ball clay showed alternate phases of reduced and increased shrinkage, as also did the kaolins in presence of aluminium chloride with ammonia. The only clay which showed a marked increase in strength in the dry state after adding aluminium chloride was the Carolina kaolin, and this effect was destroyed by ammonia; the dry strength of the ball clay was decreased by aluminium chloride. These effects are attributed to the aluminium chloride in solution and to the acidity produced by its dissociation. If larger amounts of aluminium hydroxide are present, there may be a gain in strength due to the colloidal precipitate formed.—A. B. S.

*Grog; Influence of grog in admixture with —.* W. C. Hancock. Trans. Ceram. Soc., 1919-20, 19, 149—152.

SMALL test-pieces were moulded both from a neat Stourbridge plastic clay and also from the clay mixed with grog, and were weighed at once. After drying in the air and in an oven at 105° C. they were again weighed, and the amount of tempering water thus determined. Less water was required when grog was present than for the neat clay. The amount of tempering water decreased as the percentage of grog of a particular grade increased, but increased when finer-grained grog replaced the coarser grog. In another set of experiments it was found that the finer the grog the more water was attached to it, but the amount of water attached to a particular grade seemed to diminish as the ratio of grog to clay increased. The discrepancy between the figures in the two cases was attributed to the latter mixtures being less thoroughly worked and stored for a much shorter time before moulding than the former.—H. S. H.

*Contraction of some quaternary [ceramic] mixtures fired to different temperatures.* H. S. Newman. Trans. Ceram. Soc., 1919-20, 19, 132—139.

SLABS were prepared from ball clay, china clay, stone, and flint in varying proportions, and some of each batch were fired in a saggar to cones 1a, 6a, and 8. Half were then dipped in a lead glaze and half in a leadless glaze and fired to cone 3a. The contraction at cone 016 was less in the majority



of cases than the dry contraction, showing that at a certain temperature there is an expansion, probably due to the liberation of combined water. Until the proportion of stone to flint falls below 3:2 the replacing of ball clay by china clay raises the contraction, and after this proportion is passed the reverse action takes place at the temperature of cone 1a. For cones 6a and 8 the proportion of stone to flint must be below 2:3 before the reversion occurs. The contraction does not always rise with the temperature, but with some mixtures it reaches a maximum and then falls again. The gradual substitution of one clay for the other, with the other materials remaining constant, does not have a direct bearing on the contraction, nor does the change from stone to flint whilst keeping the clays constant. A large variation may be made in the proportions of ingredients used without affecting the working properties or causing crazing or peeling.—H. S. H.

*Porcelain body; Time and temperature of burning as factors influencing the constitution and micro-structure of —.* A. A. Klein. J. Amer. Ceram. Soc., 1920, 3, 978—983.

FURTHER results, confirming those in a previous paper (J., 1916, 1220; cf. Mellor, J., 1917, 550), are given to show the effect of high temperatures in determining the constitution and microstructure of porcelain. Several samples of green table ware were burned at cone 13—14 (1348° C. by pyrometer) with a soaking period of 12—18 hrs., some of the samples being re-fired five times, i.e., with soaking periods varying from 12—18 hrs. to 72—108 hrs. One sample was fired in a pot-furnace to cone 15 for 1 hr. After one firing the felspar had melted completely, the kaolin had completely dissociated, forming amorphous sillimanite and an extremely small amount of fine crystalline sillimanite, and the solution of quartz was insignificant. After six firings there was a small increase in the amount of crystalline sillimanite and a decrease in the amount of quartz from 30 to 15%, the quartz grains being gradually rounded by solution. The sample fired for 1 hr. at cone 15 contained no amorphous sillimanite, but relatively large crystals of that mineral. Only the larger quartz crystals remained, these being well rounded, and only 6% of free quartz was left. The results show that, for the particular body used, the actual temperature of burning has a much more marked effect than the time of soaking on the constitution and structure of the porcelain.

—A. B. S.

*Lime in earthenware bodies.* A. Heath and A. Leese. Trans. Ceram. Soc., 1919-20, 19, 93—105.

PLATES were made from a body consisting of ball clay (30), china clay (25), flint (30), stone (8.25), and whiting (6.75%), and fired in a works kiln. The lime and stone were ground together. The loss from crooked, cracked, etc. ware was less than for ordinary earthenware fired at the same time. On treating with ordinary glaze and firing, the plates all crazed, harder firing producing worse effects. The contraction was less than for the body without lime, so that the lime does not act as a flux at the temperature reached. No sign of crazing was found (even when the whole of the stone was replaced by whiting) on using a glaze of the composition, (K<sub>2</sub>O 0.047, Na<sub>2</sub>O 0.267, CaO 0.449, PbO 0.237), 0.533 Al<sub>2</sub>O<sub>3</sub>, (4.3 SiO<sub>2</sub>, 0.983 B<sub>2</sub>O<sub>3</sub>).—H. S. H.

*Ceramic ware; New method for use in firing —.* K. M. Bailey. Chem.-Zeit., 1921, 45, 75.

HOT filtered air or other gas is passed into saggars or muffles so as to maintain a pressure of about 5 mm. water column therein, air or oxygen being used for oxidising, nitrogen or carbon dioxide for neutral, and hydrogen or carbon monoxide for reducing atmospheres. By this means coal of any

kind may be used, as sulphurous fumes etc. cannot reach the goods.—A. B. S.

*Aventurine glazes.* H. G. Schurecht. J. Amer. Ceram. Soc., 1920, 3, 970—977.

GLAZES of the composition 0.4 Na<sub>2</sub>O, 0.6 PbO (0.05 Al<sub>2</sub>O<sub>3</sub>, 0.25 B<sub>2</sub>O<sub>3</sub>, 0.12—0.81 Fe<sub>2</sub>O<sub>3</sub>), 2.4—4.2 SiO<sub>2</sub>, were applied to white ware fired to cone 4 under oxidising conditions. The best crystalline glazes were obtained when more than 0.41 Fe<sub>2</sub>O<sub>3</sub> was employed in glazes with 2.4 SiO<sub>2</sub>, and more than 0.58 Fe<sub>2</sub>O<sub>3</sub> in glazes with 4.2 SiO<sub>2</sub>. A variety of colours from red to brown and black may be obtained by varying the kiln atmosphere from oxidising to reducing, but if the ware is not protected in saggars the glazes become dull black owing to the formation of a ferrous lustre. If such dull glazes are covered with a suitable raw lead glaze and fired in an oxidising atmosphere, a maroon colour is obtained. The size of the crystals and the refractoriness of the glaze are increased by increasing the iron content and by slow cooling.

—A. B. S.

*Enamels for sheet steel; Classification of —.* R. R. Danielson. J. Amer. Ceram. Soc., 1920, 3, 961—970.

THE compositions of three gray ware enamels, three ground coats, three cover enamels, and two frits for colours—all of which are of proved value—are given.—A. B. S.

#### PATENTS.

*Tunnel kiln; Continuous —.* P. A. Meehan, Assr. to American Dresser Tunnel Kilns, Inc. U.S.P. 1,355,407, 12.10.20. Appl., 2.1.20.

A CONTINUOUS tunnel kiln is composed of two sections meeting at an angle. A turntable is provided within the kiln at the junction of the two parts to rotate a truck carrying goods from one section into the other.—W. F. F.

*Firebricks and refractory linings; Composition of matter for —.* A. F. Quin and C. Lacey. U.S.P. 1,355,689, 12.10.20. Appl., 5.1.20.

A REFRACTORY composition comprises ground mica, asbestos, and salt.—H. S. H.

*Pottery-ware [; Ornamenting —].* A. P. Morris. U.S.P. 1,362,956, 21.12.20. Appl., 18.12.20.

SEE E.P. 155,332 of 1919; J., 1921, 82 A.

*Refractory material.* L. Denis. U.S.P. 1,365,230, 11.1.21. Appl., 4.4.19.

SEE E.P. 144,359 of 1919; J., 1920, 546 A.

#### IX.—BUILDING MATERIALS.

*Gypsum products [plaster]; Results of testing —.* W. E. Emley and C. F. Faxon. J. Amer. Ceram. Soc., 1920, 3, 984—993.

FORTY-THREE samples made by three different manufacturers were tested according to the specification of the Amer. Soc. for Testing Materials C-26—19 T. The results of chemical analyses, calculated to CaSO<sub>4</sub>·½H<sub>2</sub>O, usually showed an excess of lime or sulphuric acid, indicating the presence of foreign material. The normal consistency or number of c.c. of water required to be added to 100 g. of dry material to produce a paste of standard "wetness" (i.e., such that the final radius of the pat in a Southard viscosimeter was 9.6 cm.) was determined. The time of set was measured by means of a Vicat needle; the temperature-rise method was found to be misleading, whereas the Vicat needle gave definite results. The fineness was determined by passing the dry material through Nos. 8 and 14

sieves, and washing it with kerosene through Nos. 28, 48, 100, and 200 sieves. The compressive strength was determined on 3 cylinders, each 2 in. diameter and 4 in. high, made of paste of normal consistency, removed from the moulds as soon as stiff enough to handle, and stored in a room for a week before testing. The yield of plaster was estimated by weighing the moulds when empty and again immediately after filling; the cylinders were also weighed just before crushing, and the weight per cb. ft. of paste, the weight of dry material per cb. ft. of paste, and the weight per cb. ft. of set material were calculated. The tensile strength was determined on briquettes of the usual form made and stored like those used for the compression test. The averages of the results obtained are:—

Character of sample.	Degree of fineness.			Compressive strength, lb. per sq. in.	Tensile strength, lb. per sq. in.	Consistency.	lb. set matter per cb. ft.	Time of set, mins.
	On 100 sieve.	100-200.	Through 200 sieve.					
Calced gypsum ..	10-1	14-5	75-1	1865	325	53	80	13
do. + retarder ..	10-5	13-1	76-3	1010	215	61	76	6:41
do. + retarder + fibre ..	11-0	15-9	73-2	1116	278	56	79	14:39
do. + retarder + wood fibre ..	20-3	13-5	66-4	835	214	57	79	9:11
do. + retarder + sand ..	64-4	10-2	25-2	335	80	25	109	2:30
do. + retarder + fibre + sand ..	64-0	8-7	27-3	415	96	26	106	3:23

The compressive strength=tensile strength $\times 4\cdot6$ .  
The tensile strength=gypsum content $\times 5\cdot3$ .

—A. B. S.

#### PATENT.

*Building block.* F. J. Kramer, Assr. to J. Whitaker. U.S.P. 1,363,045, 21.12.20. Appl., 25.5.20.

A BUILDING material consists of furnace slag pulverised and compounded with cement, together with potassium permanganate and hydrofluoric acid.

—H. S. H.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Steels; Relation of heat-treatment to cold-work in hypo-eutectoid and eutectoid —.* A. T. Adam. Iron and Steel Inst., Carnegie Scholarship Mem., 1920, 65—127.

THE experiments described were undertaken to study the processes used in wire-drawing practice, and were confined to this form of cold-work. Most of the experiments were carried out on two steels, one a basic open-hearth steel containing C 0·44%, Si 0·06%, Mn 0·82%, S 0·036%, P 0·031%; the other an acid open-hearth Swedish steel containing C 0·85%, Si 0·12%, Mn 0·30%, S 0·022%, and P 0·02%. To determine the effect of heat treatment in preparation for cold-work the steel, in the form of 4 or 5 S.W.G. rod, was heated to 850°, 950°, or 1050° C. and then cooled in air or quenched in lead at 500° or in oil and reheated to 600° C. In the case of the basic open-hearth steel the capacity to withstand cold-work increased with the temperature with air-cooling. With lead quenching lower temperatures gave better results, but the differences due to temperature were so slight that it may be concluded that the safe working range of temperature in the "patenting" process is extremely wide. Experiments with other steels confirmed this, and indicated that one of the objects of "patenting" is to enlarge the grain rather than to refine it. A good wire is produced from what would normally be an over-heated steel. With steel of eutectoid composition, i.e., of about the composition of the above Swedish steel, there is no advantage in "patenting" beyond 950° C. The improvement obtained by quenching in lead is more

marked in these steels than in medium steel. Heating below the critical range does not remove the effects of previous cold-work and cannot replace "patenting." Experiments on the effect of heat treatment after cold-work showed that hot galvanising has a had effect on the physical properties of the wire, and the higher the tenacity of the wire, the more serious is the effect. Time is an important factor, especially as regards its influence on the maximum load. The temperature of the galvanising bath should be as low as possible and the immersion should be as short as possible, consistent with the production of a satisfactory coating of zinc. The work on the microstructure showed that a fine-grained structure is unsuitable for wire-drawing. The most suitable structure is one in

which the grains are large enough to elongate into fibres and which consists throughout of granular or cellular sorbite. A structure of laminated pearlite, as in annealed eutectoid steels, is unsuitable. The structural effects of heat treatment after cold-work are scarcely visible until recrystallisation commences. When this occurs the ferrite forms new grain boundaries and the cementite collects in the form of nodules which tend to coalesce.

—E. H. R.

*Passivity. Researches on iron and nickel.* C. A. Lobry de Bruyn. Rec. Trav. Chim., 1921, 40, 30—61.

THE author finds for the equilibrium potential of iron in ferrous sulphate solution, when compared against a calomel normal electrode the value  $E_{Fe} = -0\cdot434$  volt. In connexion with the anodic polarisation of iron a graph is given showing the variation in the potential of iron electrodes in solutions of ferrous sulphate and ferrous chloride respectively as measured against a calomel electrode at different current densities. In ferrous sulphate solution when the current density reached about 0·4 ampère per sq. cm., the electrode suddenly became passive and any further increase in current density only resulted in a very feeble increase in potential. In ferrous chloride solution this sudden change was not observed, and even at the highest densities used the electrode remained active. In the passage into solution from an iron anode the current yield is practically 100% when the iron is in the active state. When, however, it is in the passive state the yield is only of the order of 1% and varies with the sample of iron used. The potential of passive iron during anodic polarisation falls as the temperature rises, and curves are given showing the activation of iron under different conditions after anodic polarisation.—W. G.

*Manganese steel; Magnetic mechanical analysis of —.* R. Hadfield, S. R. Williams, and I. S. Bowen. Proc. Roy. Soc., 1920, A, 98, 297—302.

RODS of manganese steel containing C 1·25%, Si 0·43%, and Mn 12·20%, when heated to 1000° C. and quenched in cold water, do not exhibit the Joule effect, that is they do not lengthen when subjected to a magnetic field, nor do they exhibit the Villari effect, that is, change their intensity of mag-

netisation when under tension. A slight magnetic susceptibility is exhibited if the oxide skin is not removed, but when this is removed the bars have no susceptibility. Similar bars which have been subjected to a double annealing process (heated in welded iron tubes to 550° C., allowed to cool to 450° C. in 8 hrs., and then heated at 500° C. for 60 hrs.), exhibit both the Joule and the Villari effects. The increase in length is continuous with fields varying from zero to 3000 gauss, and with the last-named field amounts to  $82 \times 10^{-4}$  cm. The intensity of magnetisation increases with the application of tension for all fields up to 3000 gauss.

—J. F. S.

*Iron and uranium; Alloys of* — E. P. Polushkin. Iron and Steel Inst., Carnegie Scholarship Mem., 1920, 129–150. (*Cf.* J., 1920, 692 A.)

FERRO-URANIUM was made in an electric arc furnace lined with a mixture of  $U_3O_8$  and pitch, and having a water-cooled bottom. The charge consisted of uranium oxide, petroleum coke, and steel turnings with fluorapatite added to form a slag. The alloys used for making uranium steel generally contain 35–45% U and 1–5% C, but alloys containing up to 90% U were prepared and examined. The alloys are generally well crystallised, are hard and pyrophoric, especially those containing higher proportions of uranium. Alloys with a high silicon (16–18%) and low carbon content are not pyrophoric, even when containing as much as 40% U. The specific gravity varies from 7.34 with 41% U to about 12.0 with 90.0% U. The melting points could not be determined with accuracy on account of oxidation of the uranium, but in the case of alloys with 28.7% and 55.2% U the m.p. was in the neighbourhood of 1540°–1660° C. in the first case and 1660°–1785° C. in the second. The alloys containing more than 40% U are decomposed by water; alloys with 40–55% U are only decomposed if their carbon content is less than 3%, but not lower than 0.7%, but alloys with 85–90% U are decomposed though their carbon content may vary from 1.2 to 7.4%. Ferro-uranium dissolves very readily in steel and becomes uniformly distributed, whether added in the furnace or in the ladle. The recovery is best when the ferro-uranium is added in the ladle. In all uranium steels part of the uranium is present as oxide. The microstructure of the ferro-uranium alloys is very complex, since five components are always present, iron, uranium, carbon, vanadium, and silicon. From the study of a series of fifty alloys the following constituents were recognised and their composition established: Metallic uranium (gamma), carbide UC (alpha), double carbide  $Fe_3C_2U_2C_3$  (iota), carbide  $U_3C_4$ , double carbide of alpha and iota,  $Fe_3U$  (beta), and  $U_3O_8$ . In addition, three conglomerates of eutectic character were found—gamma+ferrite, alpha+ferrite, and beta+ $Fe_3C$ . The constituents containing no uranium are ferrite, pearlite, V<sub>2</sub>C, FeSi, and graphite. The following table gives the principal types of microstructure:—

Main components.	Uranium. %	Carbon. %
Fe, U + uranium + ferrite .. ..	54–39	0.18–1.13
Fe, U + UC + ferrite .. ..	31–28	0.34–0.83
Ferrite + UC .. ..	20	1.10
Ferrite + Fe <sub>3</sub> U .. ..	17–5	0.53–0.76
Ferrite + pearlite + UC (steels) ..	7–0.20	0.20–0.50
Fe <sub>3</sub> C, U <sub>3</sub> C <sub>4</sub> + pearlite + FeSi ..	45–33	4.30–5.0
Fe <sub>3</sub> C, UC <sub>2</sub> + Fe <sub>3</sub> U + FeSi ..	64–48	4.90–6.1
UC + Fe <sub>3</sub> C, U <sub>3</sub> C <sub>4</sub> + Fe <sub>3</sub> U + ferrite ..	46–41	2.30–3.50
Fe <sub>3</sub> U + Fe <sub>3</sub> C + pearlite .. ..	17–4	3.6–4.3

Neither uranium nor any of its compounds mentioned above forms a solid solution with iron. The

carbide UC does not dissolve in iron even at 1200°–1250° C. The affinity of vanadium for carbon is greater than that of uranium.—E. H. R.

*Aluminium; Density of* — from 20° to 1000° C. J. D. Edwards and T. A. Moormann. Chem. and Met. Eng., 1921, 24, 61–64.

THE density of annealed aluminium (99.75% Al) was found to be 2.703 g. per c.c. at 20° C., and that of liquid aluminium at its melting point (658.7° C.) 2.332, while at higher temperatures the density is given by the equation  $D = 2.382 - [0.000272(t - 658)]$ . Tables are given showing the density of molten aluminium of different degrees of purity at various temperatures up to 1000° C. The shrinkage of solid aluminium from 658° C. to room temperature was found to be 0.21 in. per ft., while the shrinkage which takes place on solidification is approximately 6.6%. (*Cf.* J.C.S., Feb.)—A. R. P.

*Cadmium; Production of electrolytic* — H. R. Hanley. Chem. and Met. Eng., 1920, 23, 1257–1264.

A product from the bag-house of a copper smelting plant, containing approx. 0.55% Cd, 19% Zn, and 6.6% As, besides other metals, was leached with sulphuric acid, and a solution obtained, containing Zn, 96.0; Cu, 8.0; Cd, 5.0; Fe, 2.0; Mn, 0.04; As, 0.5; Co, 0.015; and Ni, 0.012 g.p.l., with traces of Bi, Ti, and Te. Treatment of this solution with powdered limestone, under air agitation, precipitated iron and traces of arsenic, and 40% of the copper, and by addition of zinc, a copper-cadmium-zinc mud, with 25% Cu, 25% Cd, and 30% Zn, was obtained which was employed as raw material for production of cadmium. The wet mud was leached with dilute sulphuric acid at 60° C., after which limestone, and then hydrated lime, were added to neutralise acidity, and the copper residue was separated. Cadmium was precipitated from the filtrate as a voluminous sponge on zinc sheets, and this, after washing, was dissolved in spent cadmium electrolyte. Iron and thallium must be removed from this solution by means of hydrated lime and sodium dichromate, respectively. When the original copper-cadmium-zinc mud contained arsenic, the zinc-cadmium solution was treated with ferric sulphate before precipitation of the cadmium sponge. For the electrolysis of the cadmium solution, double anodes of semi-circular shape were found suitable, and rotating disc cathodes of aluminium were employed. Impurities in the electrolyte must be avoided as they give rise to formation of sponge and "trees" in the cadmium deposit, and further tend to cause its corrosion. The deposited cadmium was melted under heavy oil and cast into bars, and these were re-melted under caustic soda and cast to form sticks. Cadmium has found application in the manufacture of solder, and, as a rust-preventive coating, is superior to nickel.—W. J. W.

*Hydrogenating metals; Preparation of active* — A. Brochet. Bull. Soc. Chim., 1920, 27, 897–898.

AN activated nickel may readily be prepared by heating certain of its organic salts, e.g., the formate to 200°–250° C. or the oxalate to 320°–380° C. So prepared the nickel is not spontaneously pyrophoric in the air at the ordinary temperature. Active cobalt and iron may be similarly prepared.—W. G.

*Zirconium minerals; Analysis of* — H. V. Thompson. Trans. Ceram. Soc., 1919–20, 19, 153–157.

THE mineral is fused with pure sodium peroxide in a nickel crucible; after decomposition of the melt with water the residue contains all the iron, titanium, and zirconium oxides, together with some of the silica, while the whole of the alumina and

the remainder of the silica are contained in the filtrate. Analytical details are given for the separation.—H. S. H.

*Basic slag.* Bainbridge. See XVI.

*Gold-palladium alloy crucibles.* Washington. See XXIII.

#### PATENTS.

*Alloy of iron; Rust-resisting* —. G. H. Charls. U.S.P. 1,363,584, 28.12.20. Appl., 30.6.20.

IRON is alloyed with small amounts of copper and molybdenum.—B. M. V.

*Iron; Rust-resisting alloy of* —. *Method of enhancing rust-resisting qualities of iron.* G. H. Charls. U.S.P. (A) 1,355,589 and (a) 1,355,590, 12.10.20. Appl., 7.4.20.

(A) A RUST-RESISTING iron alloy contains a small amount of molybdenum and not more than 0.2% C. (a) The rust-resisting properties of iron are enhanced by adding to the molten metal not more than 1% Mo.—A. R. P.

*Tungsten powder; Method for the production of metallic* — *direct from sodium and potassium tungstates.* C. J. Head. E.P. 155,600, 13.10.17.

FINELY ground sodium tungstate is mixed with ammonium chloride or any chloride of the iron or manganese group and a suitable reducing agent, e.g. wood charcoal, anthracite, or, preferably, sawdust. The mixture is heated in a tall nickel-chromium crucible, made in sections and set in a brick-lined, iron-sheathed furnace, at 850°–950° C. for 3–5 hrs., or until all the ammonia has been expelled. The lower half of the crucible, containing the charge is then removed and heated to 1000°–1150° C. out of contact with air, whereby the tungstic acid formed in the first reaction is reduced to metallic tungsten powder. The melt is quenched in water and the insoluble material washed in the usual way and graded to give a very pure product. The liquors from the extraction and washing of the product are treated with calcium chloride to recover any soluble unreduced tungsten, and the resulting calcium tungstate is added to the next charge in quantities not exceeding 10%. Means are provided for collecting the ammonia evolved in hydrochloric acid, the resulting ammonium chloride being used over again in the process.—A. R. P.

*Platinum and similar metals; Process for the extraction of* — *from their sands and ores.* R. Thayer. U.S.P. 1,355,186, 12.10.20. Appl., 23.1.20.

THE metals are volatilised as fume, caught in a liquid, and recovered from the liquid by electrolysis.—B. M. V.

*Alloys; Method of forming* —. P. H. Brace, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,355,532, 12.10.20. Appl., 18.4.19.

OXIDES of the metals to be alloyed are mixed, reduced with carbon to form carbides, and then melted *in vacuo* with the oxide of one of the metals.—B. M. V.

*Silver ores; Method of treating* —. L. P. Burrows. U.S.P. 1,355,795, 12.10.20. Appl., 5.12.16. Renewed 1.11.19.

CRUSHED silver ore is treated at a red heat with superheated steam, then smelted, when the different metals in the ore are found in a purified condition and in separate layers in the cooled product.—A. R. P.

*Melting metallic masses; Method of and furnace for* —. T. W. Muckle. U.S.P. 1,363,188, 21.12.20. Appl., 22.4.19.

A MELTING chamber is filled with the metal to be melted and a series of blast flames are admitted, but only after combustion has been completed outside the chamber. The hot gases permeate the metallic mass and are then exhausted through a regenerator.—B. M. V.

*Manganese or alloys of manganese; Method for producing* —. M. S. Kalling and S. D. Danielli, Assrs. to Akt. Ferrolegeringar. U.S.P. 1,363,657, 28.12.20. Appl., 4.11.19.

A MANGANESE alloy comparatively rich in silicon is exposed to free oxygen at a temperature above its melting point, e.g., by blowing with air, in order to obtain an alloy low in carbon and silicon.—B. M. V.

*Electroplating; Process of* —. J. S. Groff. U.S.P. 1,364,051, 28.12.20. Appl., 18.8.20.

A PROTECTIVE coating against corrosion is deposited on steel by making it the cathode in an electrolyte of lead fluoroborate using an anode of lead and tin; the electrolysis is continued until the proportion of tin to lead in the electrolyte is substantially the same as in the anode.—D. F. T.

*Suspensions [of ores etc.]; Process for the treatment of* —. A. Nathansohn. G.P. 323,836, 19.12.19.

SUBSTANCES sensitive to light, e.g., dyes, compounds of uranium, chromium, silver, iron, mercury, etc., or organic compounds, preferably fluorescent, such as aesculin, quinine, fluorescent dyes, or organic acids, are added to suspensions of ores or the like in suitable liquids, e.g., water, hydrocarbons, etc., and the suspension then exposed to the action of light. By this treatment the electrical charge on the suspended particles is modified and they are rendered more amenable to treatment by sedimentation or electro-osmotic processes or by magnetic separation.—A. R. P.

*Steel; Process of making alloy* —. A. Kiscock. E.P. 131,877, 23.6.19. Conv., 28.8.18.

SEE U.S.P. 1,300,279 of 1919; J., 1919, 504 A. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 3477 of 1872, 1192 and 3043 of 1875, and 793 of 1879, and in pursuance of Sect. 8, Sub-sect. 2, to E.P. 131,896.)

*Manganese or alloys of manganese; Production of* —. Aktiebolaget Ferrolegeringar. E.P. 135,186, 10.11.19. Conv., 12.11.18.

SEE U.S.P. 1,363,657 of 1920; preceding.

*Waste tins; Treatment of* —. E. J. Lovegrove. U.S.P. 1,363,332, 28.12.20. Appl., 9.8.17.

SEE E.P. 109,406 of 1917; J., 1917, 1101.

*Alloys; Method of making* —. A. G. Mumford. U.S.P. 1,355,769, 12.10.20. Appl., 26.1.20.

SEE E.P. 138,228 of 1919; J., 1920, 269 A.

*Corrosion; Prevention of* —. M. A. Adam. U.S.P. 1,365,141, 11.1.21. Appl., 31.8.20.

SEE E.P. 153,616 of 1919; J., 1921, 15 A.

*Crucible smelting-furnace.* O. M. Stein, Assr. to Soc. Anon. des Appareils de Manutention et Fours Stein. U.S.P. 1,365,204, 11.1.21. Appl., 21.5.19.

SEE E.P. 109,243 of 1916; J., 1918, 8 A.

**XL—ELECTRO-CHEMISTRY.**

*Batteries; Electric dry* —. F. Kainz. Chem.-Zeit., 1921, 45, 51—52.

THE author describes the construction of dry batteries and discusses their efficiency; the latter is small and the batteries are extremely wasteful. —W. P. S.

*Electric furnace for arsenic determination.* Birckenbach. See XXIII.

**PATENTS.**

*Ozone; Manufacture of* —. Soc. l'Azote Français, Assees. of F. Gros et Bouchardy. E.P. 140,777, 22.3.20. Conv., 22.3.19.

THE yield of ozone by subjecting air or oxygen to the action of the silent electric discharge is increased by operating so that the gas under treatment is under a pressure of 400–150 mm. of mercury, and at a temperature below  $-10^{\circ}\text{C}$ .

—J. S. G. T.

*Ozonising apparatus.* Ozoniser. W. G. Lindemann, Assr. to Ozone Co. of America. U.S.P. (a) 1,362,999 and (s) 1,363,000, 21.12.20. Appl., 15.2 and 4.8.19.

(A) A SUPPLY of ozonised water to a storage tank is automatically controlled by the water level in the tank in such manner that the ozonising apparatus operates only when large supplies of ozonised water are required. (B) An ozonising apparatus comprises an air conduit provided with inlet and discharge openings, across which a number of flat ozonising elements are arranged in a row, and spaced from the ends of the conduit. The elements engage with contact plates which are electrically connected with other contact plates mounted on longitudinal guide strips carried by a closed cylindrical metal casing.

—J. S. G. T.

*Electrolytic tanks with diaphragm cells.* G. Haglund. E.P. 151,260, 27.8.20. Conv., 17.9.19.

AN outlet whereby liquid can pass from the diaphragm cell without coming into contact with the liquid in the tank outside the cell, comprises a tube passing through the wall of the cell and making a fluid-tight connexion with a hole in the wall of the outer vessel. The tube is provided with a conical plug and is pressed against the opening in the wall of the outer vessel by a removable wedge acting on the frame of the cell. —J. S. G. T.

*Electric storage battery plates; Paste for* —. T. A. Willard. E.P. 155,944, 17.10.19.

THE active material for the plates, more especially the negative plates, of storage batteries using separators formed of a material other than wood, is mixed with 0.6% by weight of wood dust.

—J. S. G. T.

*Electrolytic cell.* H. I. Allen, Assr. to Electron Chemical Co. U.S.P. 1,355,116, 12.10.20. Appl., 5.2.18.

THE cover of an electrolytic cell is formed in sections, and carbon conductors projecting upwards from the anode pass through apertures at the ends of the sections. —J. S. G. T.

*Gases; Apparatus for treatment of* — by electric discharge. E. E. Werner. U.S.P. 1,363,827, 28.12.20. Appl., 4.3.20.

AN arc discharge is produced within a chamber having walls converging upwards. The orifice of an air nozzle located below the arcing ends of the electrodes diverges outwards and upwards.

—J. S. G. T.

*Electric batteries; Process of preparing negative plates for* —. A. Pouchain. U.S.P. 1,364,953, 11.1.21. Appl., 18.9.19.

SEE E.P. 150,810 of 1919; J., 1920, 726 A.

*Electrolysis.* M. A. Adam, J. Stevenson, A. T. Mabbitt, and J. Fieldhouse. U.S.P. 1,365,140, 11.1.21. Appl., 31.8.20.

SEE E.P. 154,635 of 1919; J., 1921, 49 A.

*Electroplating.* U.S.P. 1,364,051. See X.

**XII.—FATS; OILS; WAXES.**

*Fats and oils; Determination of water in* —. H. Oertel. Chem.-Zeit., 1921, 45, 64.

IN a method described previously (J., 1920, 824 A), oils containing more than 3% of water should be mixed with petroleum (oil, 4, petroleum, 16 g.) before the test is applied. Fats in any case required to be dissolved in petroleum, 18 g. of petroleum being used for 2 g. of fat. —W. P. S.

*Hydrogenating metals; Preparation of active* — in liquid media [oils]. A. Brochet. Bull. Soc. Chim., 1920, 27, 899—901.

IF nickel formate is heated in cottonseed oil at temperatures below  $150^{\circ}\text{C}$ . no change occurs, but at temperatures from  $160^{\circ}$  to  $270^{\circ}\text{C}$ . the formate is decomposed and the oil undergoes partial hydrogenation. The nickel is left in an active state and will complete the hydrogenation of this and more oil in an atmosphere of hydrogen under pressure at  $100^{\circ}\text{C}$ . —W. G.

*Viscosity of oils.* Schwedhelm. See IIA.

*Hydrogenating metals.* Brochet. See X.

**PATENTS.**

*Oils and fats; Apparatus for refining* — whereby they are deodorised and their acidity is reduced. K. H. Vakil. E.P. 154,514, 26.2.20.

IN an apparatus for carrying out the process described in E.P. 155,020 (J., 1921, 90 A), the crude oil flows from a tank through a heat exchanger and an electric heater to a tower, wherein it meets an ascending current of gas (carbon dioxide or a mixture of carbon dioxide and nitrogen), which may be heated if necessary by a gas heater. The gases issuing from the tower pass through a separator wherein oil mechanically carried over is separated, and the gases are then cooled by incoming gas in a heat exchanger. The still hot gases thus deprived of easily condensable fatty acids, then meet with a jet of steam and are carried to a water-cooled condenser wherein a further condensation of impurities takes place. The gases are then purified in a scrubber by means of mineral or vegetable oil and either led to a gas holder or used for purifying a further quantity of oil as described. —A. de W.

*Fat, wax, oil, gelatin, and the like; Process and apparatus for extracting* —. E. Scott and Co., Ltd., and J. Macgregor. E.P. 155,863, 17.7.19.

THE material to be extracted, e.g., the flesh or blubber of whales, seals, etc., is introduced into a vessel having a perforated false bottom and provided with a closed inlet at the top and a closed outlet in the side above the false bottom. Steam is admitted below the perforated bottom and/or by a jet or mechanical compressor in the upper part of the vessel until the charge is heated, after which further steam is admitted through the compressor only. The liquefied fat etc. percolates through the false bottom into a receiver provided with an outlet and a steam admission pipe, and steam is returned from the lower part of the vessel to the

compressor, in order to raise its temperature and compensate for losses due to condensation, and is then again introduced into the steam circulation.

—A. de W.

*Soap powders having a high percentage of liquid fats and oils; Manufacture of* — W. J. Mellersh-Jackson. From De Nordiske Fabriker De-no-fa Aktieselskap. E.P. 155,866, 25.7.19.

Soaps manufactured in the usual way from liquid fats and oils, especially drying oils and fish oils, are concentrated to a condition in which they are easy to grind and preserve, by heating in a closed boiler to 180°–200° C. at a pressure of 15–20 atm. and distilling off 10–25% of the water whilst maintaining the pressure. The soap is thereby maintained in a liquid condition during concentration, and polymerisation of the unsaturated acids readily takes place.—A. de W.

*Production of fat.* G.P. 310,616. See XVII.

*Cooling edible fats.* E.P. 155,477. See XIX.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### PATENTS.

*Zinc sulphide; Manufacture of anhydrous* —. *Manufacture of anhydrous zinc sulphide adapted for use as pigment.* P. Desachy. E.P. (a) 126,625 and (b) 126,627, 5.5.19. Conv., 19.10.17.

(A) SOLUTIONS of alkali or alkaline-earth sulphides are caused to react with hydrated zinc oxide, zinc oxide, or an insoluble zinc salt of an organic or inorganic acid to produce hydrated zinc sulphide. Alternatively zinc oxide or an insoluble zinc salt is precipitated hot or cold from a solution of zinc sulphate by means of caustic soda or sodium sulphite, the supernatant liquor containing sodium sulphate being drawn off and treated with a solution of barium sulphide. The resulting sodium sulphide solution is added gradually with the aid of a jet of steam to the zinc oxide or insoluble zinc salt, which has been previously washed, in order to convert the zinc into hydrated zinc sulphide. The latter is filtered off, washed, dried at 100° C., and calcined at 450°–500° C. in the presence of sulphur in pots provided with a cover and orifice. (B) An alkali zincate is prepared by treating metallic zinc, zinc dust, zinc oxide, calcined calamine, or roasted blende reduced to a fine powder with a hot concentrated alkali solution, any lead or cadmium being precipitated when cold by addition of a saturated aqueous solution of carbon bisulphide and filtration. A current of air charged with carbon bisulphide is led into the zincate solution heated to 80°, whereby hydrated zinc sulphide is precipitated. The latter is filtered off, washed, and calcined at 450° C. in the presence of a small quantity of sulphur, with exclusion of air.

—A. de W.

*Phenols and aldehydes; Production of condensation products of* —. L. A. Jaloustro, Z. Kheifetz, and M. Warchavsky. E.P. 138,061, 23.9.19.

SOLUBLE and/or fusible condensation products of phenols and aldehydes are transformed into insoluble, infusible, acid- and alkali-proof products, at ordinary atmospheric pressure and at a temperature not exceeding 120° C., by the aid of relatively large amounts of alkaline-earth bases or alkali cyanides, the latter also serving to accelerate the first phase of the condensation; e.g., 100 pts. of phenol, 10 pts. of slaked lime or 5 pts. of sodium cyanide, and 160–180 pts. of 40% formaldehyde, are heated to 90°–120° C.—A. de W.

*Phenols and aldehydes; Production of condensation products of* —. L. A. Jaloustro, Z. Kheifetz, and M. Warchavsky. E.P. 139,147, 23.9.19. Addn. to E.P. 138,061 (cf. *supra*).

THE liquid and soluble product obtained by the interaction of equal parts of phenol and 40% formaldehyde in the presence of 5% (on the weight of the phenol) of sodium salicylate or ammonium thiocyanate is mixed with 2% (on the weight of the phenol) of sodium cyanide and heated to 95°–100° C. in an open vessel to yield a solid, insoluble, infusible, acid- and alkali-proof product.—A. de W.

*Condensation products of acrolein with phenols; Process for the preparation of* —. C. Moureu and C. Dufraisse. E.P. 141,059, 31.3.20. Conv., 31.3.19.

A HARD, insoluble, non-conducting resin is produced by the condensation in a single operation of phenol and acrolein with about 1% of caustic soda. The acrolein may be replaced by a resin or polymerisation product thereof.—W. F. F.

*Linoleum; Working up of linoleum scraps into new* —. V. Scholz and C. Tiedemann. E.P. 143,561, 20.5.20. Conv., 16.5.18.

LINOLEUM scrap is heated with organic "depolymerising agents," e.g., ethylene trichloride, benzoline, and/or methylated spirit to 80°–140° C., the amount of solvent used being only sufficient to effect a softening of the scrap, whereby the adhering jute fibres may be mechanically removed. The solvent is removed from the treated scrap by evaporation, and the residual pulp heated to 100°–120° C. to regenerate linoleum and restore the qualities of toughness, glutinousness, etc., to the soft depolymerised linoleum, whereby it may be used alone or in conjunction with fresh linoleum cement for the production of linoleum.—A. de W.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Rubber; Influence of tapping on latex and* —. O. de Vries. Comm. Central Rubber Station, Buitenzorg, 1920, 4, No. 22, 313–334.

LENGTHENING of the tapping cut or increase in the number of tapping cuts on a tapping surface, has the same effect as heavier tapping generally, the sp. gr. of the latex increasing and its rubber content diminishing, whilst the rubber vulcanises more rapidly. Tapping to the wood produced a similar result, a latex being obtained on some days containing 16% of rubber and having sp. gr. 1.0002. Tapping at different hours of the day or tapping twice daily on the same cuts caused no alteration in the properties of the rubber. On tapping a piece of bark which had been isolated by cuts to the wood, the yield of latex often failed gradually, but the rubber content of the latex never approached the neighbourhood of 10%.—D. F. T.

*Rubber; Ageing of vulcanised* —. O. de Vries and H. J. Hellendoorn. Comm. Central Rubber Station, Buitenzorg, 1920, 4, No. 24, 429–457.

IN storage experiments with vulcanised rubber at the ordinary temperature the samples underwent a continuous reduction in extensibility at a greater rate than that observed earlier by Stevens; the increase in tensile strength on storage never markedly surpassed the value obtained for the same stress-strain curve by direct vulcanisation. The maxima observed in the tensile strength were much less sharp than in Stevens' experiments, and, with a vulcanisation coefficient of 4–8, the tensile strength in several cases continued to increase for 80 weeks;

inferior samples deteriorated more rapidly. A mixture with only 5% of sulphur when fully vulcanised behaved in exactly the same way as one containing a considerable proportion of free sulphur. Whereas, however, vulcanisates with 5–10% total sulphur containing uncombined sulphur, on keeping remained dry and gradually became brittle, those containing 5% of sulphur, all of which was in the combined state as a result of prolonged vulcanisation, became weak and sticky. (Cf. de Vries, J., 1919, 82 A; Stevens, J., 1918, 305 T, 340 T.)—D. F. T.

*Rubber; Influence of some factors in rubber preparation on the ageing qualities of the vulcanised* — O. de Vries and H. J. Hellendoorn. Comm. Central Rubber Stat., Buitenzorg, 1920, 4, No. 24, 458–472.

THE alteration of vulcanised rubber on storage is unaffected by "maturing" or excessive milling of the coagulum, by variation, within the usual limits, in the amount of acetic acid used for coagulation, by the use of small amounts of sulphuric acid, or by the application of sugar or salt for coagulating purposes. Coagulation by evaporation and partial coagulation yield rubbers which, like some lower grades, exhibit a more rapid deterioration with respect to tensile strength.—D. F. T.

*Rubber stress-strain curve; Some aspects of the* — W. B. Wiegand. Canadian Chem. J., 1920, 4, 160–170. Indiarubber J., 1920, 60, 379–383, 423–429.

AFTER reviewing the present state of knowledge of the physics of rubber from the point of view of the stress-strain relationship, hysteresis and thermal phenomena (Joule effect), experiments as to the comparative effect of various inorganic compounding ingredients on the stress-strain curve are described. Gradually increasing proportions of lampblack, gas black, China clay, red iron oxide, zinc oxide, glue, whitening, fossil flour, and barytes respectively were added to the standard mixing of rubber (100 vols.), litharge (3 vols.), and sulphur (2½ vols.), the mixtures being subsequently vulcanised and the "stress-strain" curve measured on a Scott machine. The toughening effect of each of the ingredients named was measured by the area included between the curve and the vertical (elongation) axis; the addition of barytes, fossil flour, glue, whitening, and red iron oxide diminishes the energy content of the stretched rubber, whereas zinc oxide, lampblack, and gas black cause an increase, the effect being greatest with the last-named. The effect of the various "fillers" runs parallel to the fineness of their particles or to the extent of the surface developed in the rubber. If the proportion of additional ingredient is raised beyond a certain limit, e.g., beyond 20 vols. of zinc oxide or 40 vols. of gas black, the particles begin to agglomerate, with consequent diminution of the ultimate tensile strength and "toughening effect."—D. F. T.

*Chinosol.* Spoon. See XIXs.

#### PATENTS.

*Vulcanised rubber.* C. R. Boggs, Assr. to Simplex Wire and Cable Co. U.S.P. 1,364,055, 28.12.20. Appl., 11.9.17.

RUBBER is mixed with inert compounding ingredients, selenium, and an aromatic amine, and vulcanised.—D. F. T.

*Rubber and other heavy plastic material; Machines for kneading or mizing* — F. H. Banbury. E.P. 156,273, 17.9.18.

## XV.—LEATHER; BONE; HORN; GLUE.

*Skin structure and bating.* A. Seymour-Jones. J. Soc. Leather Trades' Chem., 1920, 4, 291–293.

PELT consists of grain membrane, grain or "true skin," fatty layer, and the "corium." The grain membrane is packed with hair sheaths, sebaceous glands, muscles, sweat ducts, etc., placed amidst extremely fine collagenous fibres, which are collected into bundles and held in a vertical position by elastic fibres. These maintain the structural scheme of the grain and give it elasticity. They are absent in other layers. Bating removes them, and experiments show that skins should be delimed with acid, pasted on the grain with a trypsin preparation, and then submitted to some process which will remove the fat from the fatty layer. A completely satisfactory process has not yet been evolved.—D. W.

*Fat-liquoring [chrome leather]; Effect of soap in* — R. F. Innes. J. Soc. Leather Trades' Chem., 1920, 4, 299–300.

EXPERIMENTS on fat-liquoring chrome-tanned goat skin with different quantities of soap showed that an acid chromium soap was formed by the double decomposition of the soap with the basic chromium sulphate in the leather, giving potassium sulphate, which remained in the solution. Two batches of leather were fat-liquored under the same conditions with a potassium soap and a sodium soap of the same strength and quantity of total fatty acids. No difference could be detected in the finished leathers, and no potassium was found in the batch treated with potassium soap. The soap in a fat-liquored skin cannot be extracted by water, since a water-soluble soap is no longer present. The alkali in the soap merely acts as a carrier of the fatty acids. The basicity of the chromium salt on the fibres of the leather diminishes with increased amount of soap in the fat-liquor.—D. W.

*Gelatin and glue; Jelly value of* — M. Ishikawa. Kōgyō Kwagaku Zasshi (J. Chem. Ind. Tokyo), 1920, 23, 1147–1152.

THE following modification of Clark's method (J., 1918, 665 A) is proposed:—A test-tube of 1.2 cm. inner diameter, having a mark 1 in. from the bottom, is filled with the sample (2% gelatin solution or 5% glue solution) to the mark and immersed in a beaker filled with water and provided with a thermometer. The beaker is cooled with ice water during more than 1 hr., then warmed gradually to the m.p. of the sample, i.e., until the surface of the sample just forms a horizontal plane on inclining the tube.—K. K.

*Proteins and tannin.* Sollmann. See XX.

#### PATENTS.

*Tanning of chrome leather.* C. F. L. Barber and P. R. Barker. E.P. 155,887, 16.9.19.

A ON-BATH chrome tanning liquor is prepared by reducing solutions of chromic acid, or bichromate and acid, with synthetic tan. For the two-bath process a strong solution of synthetic tans is used as the reduction bath. (Reference is directed, in pursuance of Sect. 7, Subject 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 16,647 of 1886 and 287 of 1908.)—D. W.

*Tannage of hides and skins; Process and apparatus for the instantaneous* — F. Gilardini. U.S.P. 1,363,771, 28.12.20. Appl., 6.4.18. Renewed 17.11.20.

SEE E.P. 114,631 of 1918; J., 1919, 297 A.



*Waste liquors of the hide-treating art; Processes for recovery of proteids from —.* W. J. Mellersh-Jackson. From The Dorr Co. E.P. 156,444, 14.4.20.

SEE U.S.P. 1,347,822-3 of 1920; J., 1920, 633 A.

*Extracting gelatin.* E.P. 155,863. See XII.

## XVI.—SOILS; FERTILISERS.

*Potash lime, magnesia; Respective rôle of the three bases— in cultivated plants.* H. Lagatu. Comptes rend., 1921, 172, 129—131.

From the analyses of numerous plants the author has calculated the basic equivalents,  $K_2O/2$ ,  $CaO/2$ ,  $MgO/2$ , in 100 basic equivalents attributable to these three bases and plotted the results on a triangular diagram for a large number of different species. The ratio  $MgO/CaO$  is only  $>1$  in the case of sugar beet, maize, potatoes, and mangels, exactly 1 for wheat, and just under 1 for oats, rye, barley, and buckwheat.—W. G.

*Phosphates in basic slag; Effect of fluorspar additions on the —.* F. Bainbridge. Iron and Steel Inst., Carnegie Scholarship Mem., 1920, 1—40.

*Parts I. and II. Manurial value of basic slag containing fluorspar.* Continuing the work previously described (J., 1919, 649 A), experiments were made to examine the possibility of increasing the fertilising value of insoluble phosphate by adding it to the soil in the late autumn and thus exposing it during the winter to the action of the atmosphere. The results showed a bigger proportionate yield compared with a soluble slag when the insoluble phosphate was applied in the autumn instead of in spring. The insoluble slag gave satisfactory yields of both grain and straw (barley), but failed to give the increased phosphate content of grain and straw given by soluble slag.

*Part III. Nature and solubility of the phosphates contained in basic open-hearth slag made with additions of fluorspar.* In experiments to prepare synthetically the compounds presumed to be present in basic slag, uncombined lime in the fusions was determined by passing steam over a weighed quantity of the material contained in a platinum boat in a silica tube heated to  $140^{\circ}C$ , the gain in weight being attributed to the hydration of lime. Tri-basic calcium phosphate is much more soluble in 2% citric acid than in ammonium citrate solution; consequently the use of the latter solvent was abandoned. The fusion of the tribasic phosphate with calcium fluoride caused a rapid fall in the solubility of the phosphate up to a point corresponding with 8 pts.  $CaF_2$  to 100 pts. of phosphate. Tetra-basic calcium phosphate was prepared by fusing the tribasic phosphate with lime in the oxy-acetylene blowpipe flame. The product contained 94.12% of tetrabasic phosphate and 0.9% of free lime. A solubility determination showed that only 21.5% of the phosphate present was soluble when ground to 100-mesh, but 72.26% when ground to flour. The tetrabasic phosphate is decomposed when fused with calcium fluoride, apatite being formed, with the result that the solubility is lowered. Silico-carnotite,  $3CaO.P_2O_5.2CaO.SiO_2$ , was prepared by fusing dicalcium silicate with tribasic calcium phosphate, and was completely soluble in citric acid. Fusion of silico-carnotite with calcium fluoride rapidly lowered the solubility. A number of fusions of basic slag with varying quantities of calcium fluoride were made, and the products were analysed and their solubilities determined. Curves

are given showing the lowering effect on the solubility of the phosphate of increasing quantities of calcium fluoride. It is concluded that the addition of calcium fluoride to basic open-hearth slags results in the formation of an apatite of the formula  $3(3CaO.P_2O_5).CaF_2$ . Crystals which were identified as apatite were actually obtained from such a slag. In extremely soluble basic slags the phosphate is probably present as silico-carnotite or steatite. Free lime does not exist to any extent in normally prepared slag containing about 14%  $P_2O_5$ .

*Appendix. Effect of the addition of lime on the citric acid solubility of a basic slag.* The result of re-fusing a slag, without addition of lime, is to increase considerably the solubility of the phosphate present. Addition of lime does not affect appreciably the solubility of the phosphate, whilst the added lime combines, probably with the iron oxide present, to form a compound almost insoluble in citric acid.—E. H. R.

*Potash in commercial wood ashes; Availability of —.* R. E. Rose. J. Assoc. Off. Agric. Chem., 1920, 3, 323—326.

THE potassium in wood ashes is present chiefly in the form of carbonate and very little, if any, exists as silicate, and there is nothing to be gained by extracting the material with 1% citric acid solution in place of boiling water in determining the available potash.—W. P. S.

*Kjeldahl-Gunning-Arnold method for the determination of ammonia in fertilisers; Substitution of sodium sulphate for potassium sulphate in the —.* T. D. Jarrell. J. Assoc. Off. Agric. Chem., 1920, 3, 304—306.

PRACTICALLY identical results were obtained, whether sodium sulphate or potassium sulphate was used.—W. P. S.

*Sulphur; Mechanism of the fertilising action of —.* G. Nicolas. Comptes rend., 1921, 172, 85—87.

IN addition to the other rôles played by sulphur, it is shown that it favourably influences the assimilation of carbon dioxide by plants. The optimum dressing of sulphur varies with the species of plant.—W. G.

## PATENT.

*Nitrogenous phosphatic material and process of producing the same.* F. S. Washburn, Assr. to American Cyanamid Co. U.S.P. 1,355,369, 12.10.20. Appl., 23.7.20.

CRUDE calcium cyanamide is treated with water, and the resulting solution is treated with phosphoric acid.—W. J. W.

## XVII.—SUGARS; STARCHES; GUMS.

*Sugar solutions; Refraction and dispersion of —.* H. Krüss. Z. Ver. deuts. Zuckerind., 1920, 617—625.

From the values determined by Matthiesson (Diss., 1898), Main (Int. Sugar J., 1907, 9, 481), and Schönrock (Z. Instrk., 1911, 31, 191), the author has compiled a single table of refractive indices of sugar solutions at  $20^{\circ}C$ . (based on a smoothed-out curve), which is considered to be more accurate than the individual tables from which it was compiled. On the basis of this table Matthiesson's values for the refractive indices of sugar solutions for the C, D, and F lines have been corrected. The optical design of Schönrock's sugar refractometer (J., 1914, 154) is discussed.—J. H. L.

*Sugars and polyatomic alcohols; Reactions of — in boric acid and borate solutions, with some analytical applications.* G. Van B. Gilmour. *Analyst*, 1921, 46, 3—10.

THE combination of polyatomic alcohols, sugars, and many hydroxy compounds with boric acid to produce stronger acids is discussed. When an excess of mannitol is added to boric acid, mannito-boric acid is formed; this yields a sodium salt which is stable in acid solution, but decomposes in neutral or alkaline solution with the production of sodium metaborate and mannitol. The metaborate then combines with mannitol to form compounds like  $\text{NaBO}_3 \cdot 3\text{C}_6\text{H}_{11}\text{O}_5$ . This type of reaction may be considered general for the polyhydroxy compounds that permit of the volumetric determination of boric acid. Amongst the analytical applications of the reactions is the determination of levulose in invert sugar etc. A weighed amount of the latter is treated with 10 c.c. of  $M/10$  boric acid solution and 0.5 c.c. of 1% phenolphthalein solution, and the mixture is titrated with  $N/10$  sodium hydroxide solution. The quantities of levulose corresponding with the various volumes of alkali solution are as follows:—7.2 c.c., 0.22 g.; 8.1 c.c., 0.27 g.; 8.5 c.c., 0.31 g.; 8.9 c.c., 0.36 g.; 9.2 c.c., 0.40 g.; 9.5 c.c., 0.45 g.—W. P. S.

## XVIII.—FERMENTATION INDUSTRIES.

*Enzymes; Formation of —.* E. Köhler. *Biochem. Zeits.*, 1920, 112, 236—254.

THE course of fermentation depends on the formation of a pro-enzyme of zymase and on the activation of the latter. Maltose is capable of activating zymase, whilst other sugars, such as dextrose, sucrose, and levulose, have an inhibiting action. On the other hand, in the production of the pro-enzyme maltose exerts an inhibiting influence in contradistinction to the other sugars. This explains the different physiological behaviour of certain yeasts to the various sugars.—S. S. Z.

*Enzyme action; Mechanism of —.* I. Rôle of the reaction of the medium in fixing the optimum temperature of a ferment. A. Compton. *Proc. Roy. Soc.*, 1921, B, 92, 1—6.

EXPERIMENTS with a highly active maltase preparation indicate that the optimum temperature of a ferment or ferment function, occurring in a given enzyme preparation, is independent of the concentration of the enzyme, the duration of the action and the hydrogen ion concentration of the medium being constant (*cf.* Compton, J., 1914, 977).

—J. C. D.

*Peroxydases. II.* R. Willstätter and M. Bommer. *Annalen*, 1921, 422, 47—73.

SLICED horse-radish which is immersed in flowing water so that dialysis takes place through the cell walls generates new peroxydase so long as the cells retain their vitality. The peroxydase number falls during the first four days and then increases during the following two or three weeks. If the dialysing water contains toluene new peroxydase is not formed and the peroxydase number falls rapidly owing to exosmosis of the enzyme. Evidence is recorded to show that the plant material contains two peroxydases, one of which is easily soluble, the other completely insoluble, in water. During dialysis the amount of the soluble peroxydase decreases, and that of the insoluble peroxydase increases with the time, so that in the preparation of peroxydase extract the time of dialysis should not exceed 12 days. Peroxydase is almost completely adsorbed by aluminium hydroxide from a solution in 50% alcohol at a concentration of 0.05%, and 90—95% of the peroxydase is recovered in solution in a

more active condition by agitating the adsorption product with water containing carbon dioxide at 0°—20° C. On this discovery is based a modification of Willstätter and Stoll's method of preparing peroxydase extract (*Annalen*, 1918, 416, 21) whereby an active peroxydase preparation (purpurogallin number 860) is obtained from horse-radish with a great saving of time and labour. (*Cf.* J.C.S., Feb.)—C. S.

*Lactic acid; Separation and identification of —, as complex ferric-sodium lactate.* K. A. Hofmann. *Ber.*, 1920, 53, 2224—2226.

TO separate lactic acid from fermentation products the solution is mixed with little more than the estimated amount of ferric chloride, rendered alkaline with sodium carbonate, acidified with acetic acid, and evaporated on a water bath, when a characteristic very pale green precipitate of ferric-sodium lactate,  $[\text{Fe}(\text{C}_2\text{H}_3\text{O}_2)_3] \cdot \text{Na}_2\text{H}_2\text{O}$ , separates in the course of a few hours. This is almost insoluble in water at 20° C., and is only slowly attacked by dilute alkalis, 1% hydrochloric acid, 15% acetic acid, or tannic acid. (*Cf.* J.C.S., Feb.)—J. C. W.

*Glycerol in cider vinegar, Isolation and identification of —.* R. W. Balcom and E. G. Grab. *J. Assoc. Off. Agric. Chem.*, 1920, 3, 411—412.

GLYCEROL was extracted from 5 l. of cider vinegar by the official (American) method for the determination of glycerol in vinegar; the glycerol was then distilled under reduced pressure in the presence of sandalwood oil, the aqueous solution obtained, after removal of the sandalwood oil, was evaporated at a low temperature, and the glycerol dried under reduced pressure over sulphuric acid. The product thus obtained was proved to be pure glycerol by oxidation with dichromate (usual method for the determination of glycerol) and by the m.p. of its benzoyl derivative.—W. P. S.

*Papain.* Chesnut. *See XX.*

### PATENT.

*Fat from raw materials containing carbohydrates; Production of — by means of fungi.* Kriegsausschuss f. pflanzliche u. tierische Oele u. Fette G.m.b.H. G.P. 310,616, 6.6.16.

MATERIALS such as turnips, potatoes, apples, or pears in subdivided form are inoculated with fungi of the sachsia, oidium, endomyces, or yeast groups, and allowed to ferment with access of air. Either before or during fermentation an ammonium salt, urea, or sugar, may be added. When distillery yeast or "mineral yeast" is employed an oil of good quality, similar to olive oil, and containing glycerides of fatty acids with traces of free acid, is produced.—W. J. W.

## XIXa.—FOODS.

*Milk; Indirect analysis of — and the detection of added water.* A. Bouriez. *Ann. Falsif.*, 1920, 13, 606—618.

THE total solids, casein, soluble matters other than casein, sp. gr. of the serum, and sp. gr. of the fat-free milk may be calculated from the fat content and the sp. gr. of the milk. Total solids (g. per litre) =  $1.17B + 8/3 \times 1000(D-1)$ ; casein =  $0.3(T-B)$ ; soluble matters =  $0.7(T-B)$ ; sp. gr. of the serum =  $750(T-B)/2.5(1000D-T) + (T-B)$ ; and sp. gr. of the fat-free milk =  $375(T-B)/(1000D-T) + 0.625(T-B)$ , where T = total solids, B = g. of fat per litre, and D = the sp. gr. at 15° C.—W. P. S.

*Milk serum [; Specific gravity of —].* R. Ledent. *Ann. Falsif.*, 1920, 13, 601—605.

THE sp. gr. of milk serum (obtained by heating the milk at 70° C. with the addition of 20% acetic acid)

varies from 1.027 to 1.029, and is decreased to 1.023 by the addition of 10% of water to the milk. Milk from diseased cows yields a serum having a sp. gr. lower than the limit mentioned. A low sp. gr. indicates therefore the presence of added water or that the milk is from a diseased cow.—W. P. S.

*Lard; Detection of beef fat in* —. Vitoux and C. F. Muttelet. *Ann. Falsif.*, 1920, 13, 593—601.

THE method described is that proposed originally by Bömer (J., 1913, 434), and depends on the presence of  $\alpha$ -palmitodistearin, m.p. 68.5° C., in lard, and of  $\beta$ -palmitodistearin, m.p. 63.3° C., in beef fat. The fatty acids separated from these two glycerides melt at 63.2° C. 50 g. of the fat is dissolved in 50 c.c. of pure acetone, the solution is cooled to 15° C., and after 1 hr. the separated crystals are collected on a filter, dried over sulphuric acid, then dissolved in 50 c.c. of ether and again allowed to crystallise. These crystals are collected, the m.p. determined, a portion then saponified, the fatty acids separated, and their m.p. determined. The crystals of glycerides obtained from pure lard melt above 62° C., those from beef fat at about 58.5° C. In the case of pure lard the value (2G-A) is never less than 68°; G = the m. pt. of the crystals, and A = that of the fatty acids.—W. P. S.

*Antiscorbutic action of raw potatoes, crushed and whole*. Bezsonoff. *Comptes rend.*, 1921, 172, 92—94.

THE antiscorbutic action of raw peeled potatoes, not crushed, is equal to that of vegetables having a marked antiscorbutic action, such as the cabbage or dandelion, but if the potatoes are first crushed the action is much inferior. The expressed juice possesses an antiscorbutic action, but less than that of the equivalent weight of uncrushed potato, whilst the action of the marc is practically nil.—W. G.

*Proteins; Free amino groups of* —. II. S. Edlbacher. *Z. physiol. Chem.*, 1919, 108, 287—294. (Cf. J., 1920., 796 A).

THE "formol value" and the "methyl value" obtained by methylating with dimethyl sulphate were determined in gelatin and casein digested with pepsin and in gliadin and zein digested with hydrochloric acid and in some undigested proteins. The relation of the figures obtained to the free amino groups in the protein molecule is discussed. In esocin, scombrin, gliadin, and zein there appears to be a certain parallelism between the lysine content and the free amino groups. Clupein and salmin, which are free from lysine, contain a larger number of nitrogen atoms in the form of groups, probably imino groups, which can be methylated but are not indicated by formol titration. In gelatin, casein, sturin, edestin, and cyprinin, the "methyl values" indicate 3—5 CH<sub>3</sub> groups attached to nitrogen for each amino group shown by formol titration, so that each free amino group is probably converted into a N(CH<sub>3</sub>)<sub>2</sub> on methylation. Thymus- and gadus-histones, on the other hand, show a much higher content of nitrogen by formol titration than corresponds to the "methyl value."—S. S. Z.

*Histidine; Separation of* — from arginine. A. Kossel and S. Edlbacher. *Z. physiol. Chem.*, 1920, 110, 241—245.

IN the method described previously (*Z. physiol. Chem.*, 1901, 31, 171) for the separation of histidine and arginine by precipitation at different degrees of alkalinity of the medium, precipitation of histidine is complete even in absence of the more strongly alkaline arginine. The precipitation of histidine begins whilst the reaction of the medium is still acid and is complete when the solution reddens phenolphthalein. Iminazole behaves similarly, as also do carnosine and guanidine, except

that in the last two cases there is no precipitation so long as the medium is still acid or even neutral to litmus. Methylguanidine is precipitated incompletely in a medium which reddens phenolphthalein and completely when the solution gives a blue colour with thymolphthalein. Arginine is precipitated only when the solution gives a blue colour with thymolphthalein; creatinine is precipitated incompletely under the same conditions. Glycine gives a white precipitate in a medium which reddens phenolphthalein and a brown precipitate in a medium which gives a blue colour with thymolphthalein.

*Sericin and the quantitative estimation of its constituents*. W. Türk. *Z. physiol. Chem.*, 1920, 111, 71—75.

SILK was treated with water at 145° C. for 3 hrs. under a pressure of 3 atm. Sericin went into solution and was partly precipitated from it with absolute alcohol. Both the soluble and insoluble portions were examined for the various protein colour reactions. Those depending upon the presence of a carbohydrate complex in the protein molecule, e.g., the reactions with  $\alpha$ -naphthol and with thymol, were very intense, and were the same for both the soluble and insoluble portions. A mixture of 300 g. of the alcohol-insoluble sericin and 100 g. of the alcohol-soluble portion was hydrolysed with sulphuric acid and yielded (calculated to dry substance) 5.69% of tyrosine, 1.79% of leucine, 6.81% of serine, 4.56% of arginine, 1.96% of lysine, and 1.02% of histidine.—S. S. Z.

*Saccharin and dulcin*. Paul. See XX.

*Papain*. Chesnut. See XX.

#### PATENTS.

*Fats; Cooling apparatus for use in the manufacture of edible* —. W. Clayton and G. Nodder. E.P. 155,477, 6.11.19.

A SERIES of hollow disc-shaped sections are placed face to face to form the body of the cooler; a cooling liquid is passed through the interior of the sections and the fat to be cooled is forced through the narrow circular chambers between the discs. These chambers are provided with scrapers fitted to a rotating shaft passing through the centres of the discs.—W. P. S.

*Vegetables and the like; Apparatus for drying* —. W. Spoelstra. E.P. 155,625, 2.9.19.

THE material is fed on to the uppermost of a series of shelves arranged between the two walls of a rotating vertical double-walled cylindrical vessel. The shelves are divided into sectors and as the apparatus rotates each sector is tilted in turn and discharges its contents on to the shelf beneath. Hot air passes up the central chamber and through adjustable openings into the space between each pair of shelves thus supplying fresh drying air to the material on each shelf.—L. A. C.

*Drying liquids [e.g. milk]*. J. H. Akkerman. E.P. 155,927, 9.10.19.

THE liquid to be dried is applied by a spraying or like device to a rapidly rotating disc which is not heated but may be in a moisture-absorbing atmosphere; the conditions are adjusted so that the material is dry before it leaves the disc, and if it should stick there is no need to remove the dry material continually as no heat is applied to it.

—B. M. V.

*Milk; Process for making evaporated* —. O. W. Mojonier. U.S.P. 1,362,723, 21.12.20. Appl., 8.5.20.

THE milk is condensed by heat and a number of

samples are withdrawn and, after addition of varying quantities of sodium bicarbonate, are heated at 243° F. (117° C.) for 15 mins. in a steam retort with a cage revolving at a rate of 10 revs. per min. The samples are cooled with cold water, and the quantity of sodium bicarbonate required by the bulk of the milk for sterilisation is ascertained by selecting the best sample.—L. A. C.

**Fruit-juice; Process of treating —. Process of making jelly. Process of making fruit syrup.** M. O. Johnson. U.S.P. (A) 1,362,868, (a) 1,362,869, and (c) 1,362,870, 21.12.20. Appl., (A and a) 12.9.19, (c) 17.1.20.

FRUIT-JUICE is concentrated by freezing a portion of the water content and separating the residual liquid, and is then clarified by heating to a temperature high enough to coagulate suspended matter but not high enough to impair the flavour. After separation of coagulated matter the juice is (A) sterilised by heating to a temperature below that employed for coagulation, and (a) jellified after addition of sugar. In (c) sugar is added to the juice before coagulation to obtain better clarification.—L. A. C.

**Desiccating liquids.** U.S.P. 1,362,590. See I.

**Moisture in cereals.** E.P. 143,191 and 155,486. See XXXIII.

## XIXa.—WATER PURIFICATION; SANITATION.

**Arsenical insecticides; Determination of arsenious acid and arsenic acid in the presence of each other in —.** R. C. Roark. J. Assoc. Off. Agric. Chem., 1920, 3, 358—368.

To determine arsenious acid in lead arsenate a weighed portion of the sample is boiled for 30 mins. with 100 c.c. of dilute sulphuric acid, cooled, diluted to 200 c.c., and filtered; 100 c.c. of the filtrate is nearly neutralised with sodium hydroxide, excess of sodium bicarbonate is added, and the arsenious acid is titrated with N/20 iodine solution. Of 100 samples examined by the author, 15 contained 0.34—3.54% As<sub>2</sub>O<sub>3</sub>. Arsenic acid (together with antimony) is determined by a method depending on the reaction  $\text{As}_2\text{O}_5 + 4\text{HI} = \text{As}_2\text{O}_3 + 2\text{I}_2 + 2\text{H}_2\text{O}$ . A portion of the sample is treated with concentrated hydrochloric acid, the mixture evaporated to dryness, the residue dissolved in 50 c.c. of concentrated hydrochloric acid, 10 c.c. of 20% potassium iodide and 50 c.c. of 25% ammonium chloride solution are added, and the liberated iodine is titrated with thio-sulphate solution, which has been standardised against pure lead arsenate. The method of determining arsenic acid in lead arsenate may be applied to zinc arsenite, and the total arsenic in this compound may be determined by dissolving the sample in acetic acid, precipitating the zinc as oxalate, and titrating the arsenic in the filtrate after reduction with potassium iodide and sulphuric acid.

—W. P. S.

**Chinosol.** W. Spoon. Comm. Central Rubber Stat., Buitenzorg, 1920, 4, No. 22, 335—345.

CHINOSOL is sometimes added as a disinfectant to latex to prevent "spottedness" and "rustiness" in the finished rubber. As the active principle, *o*-hydroxyquinoline, is present as sulphate, frequently admixed with potassium sulphate, the value of commercial chinosol is conveniently determined from its content of *o*-hydroxyquinoline calculated from the expression  $9.05 \times (S - 0.41K)$  in which S and K represent respectively the percentage of sulphur and potassium.—D. F. T.

**Chloromethyl carbonates and chlorocarbonates; Toxicity of the —.** A. Mayer, H. Magne, and L. Plantefol. Comptes rend., 1921 172, 136—139.

THE TOXIC effect of the chloromethyl carbonates, as measured with rabbits, guinea-pigs, and dogs, increases with the successive introduction of chlorine atoms up to the trichloro stage. The introduction of a fourth chlorine atom if the first three were all in one methyl group diminishes the toxicity, but does not alter it if they were distributed between the two groups. The introduction of two further atoms of chlorine notably increases the toxicity. The most symmetrical distribution of the chlorine atoms gives the least toxicity. The chloromethyl chlorocarbonates become more and more toxic as the number of chlorine atoms increases, and for the same number of chlorine atoms present in the molecule they are more toxic than the carbonates.—W. G.

**Hydrogen cyanide.** Sieverts and Hermsdorf. See VII.

## PATENTS.

**Water-softening apparatus.** J. E. Caps. U.S.P. 1,362,673, 21.12.20. Appl., 29.3.17.

IN A WATER-SOFTENING apparatus, comprising a container for the water to be treated, a filter-bed containing water-softening agent, and a reservoir for the regenerating liquid, the variations of the water level control a mechanism which operates valves whereby the regeneration and flushing of the filter-bed, and the re-filling of the water-container, are effected consecutively.—W. J. W.

**Copper-containing compound for treating plant diseases and process of making it.** G. Morselli. U.S.P. 1,362,172, 14.12.20. Appl., 5.5.19.

CUPRIC chloride solution (46° B., sp. gr. 1.4646) is treated with a slight excess of milk-of-lime containing 15% of calcium hydroxide; the precipitate formed is collected, washed, dried, and powdered. It has the composition  $\text{CaCl}_2 \cdot 3\text{CuO} \cdot x\text{H}_2\text{O}$ .—W. P. S.

**[Water;] Process for removal of gases from liquids [—].** W. S. Elliott. E.P. 155,864, 17.7.19.

SEE U.S.P. 1,321,999 of 1919; J., 1920, 80 A.

**Distilling water; Method of and means for [on ships].** H. G. Cruikshank. From Lysekils Mekaniska Verkstads Aktiebolag. E.P. 156,450, 27.4.20.

**Disinfecting agent.** E.P. 147,535. See VII.

**Ozonising apparatus.** U.S.P. 1,362,999. See XI.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Thebaine; Reduction products of —.** M. Freund and E. Speyer. Ber., 1920, 53, 2250—2264.

A PHENOLIC dihydrothebaine has already been obtained by reducing thebaine with sodium and alcohol. Hydrogenation in cold N/1 hydrochloric acid in the presence of spongy platinum yields a new dihydrothebaine which is insoluble in sodium hydroxide, whereas a reduction product of this, namely dihydrothebainone, is formed if colloidal palladium is the catalyst. On hydrolysis with dilute mineral acids the new dihydrothebaine loses methyl alcohol and gives dihydrocodeinone. (Cf. J.C.S., Feb.)—J. C. W.

**r-Ecgonine and tropinone; Complete synthesis of —.** R. Willstätter and M. Bommer. Annalen, 1921, 422, 15—35.

**POTASSIUM ethyl acetonedicarboxylate**, obtained

from citric acid, is electrolysed in a divided cell, whereby ethyl succinyldiacetate  $C_8H_{14}(CO_2CH_2)_2CO_2$ , prisms, m.p.  $46^{\circ}$ – $47^{\circ}$  C., is obtained, which reacts with a warm, concentrated solution of methylamine acetate to give ethyl 1-methylpyrrole-2,5-diacetate, domed prisms, m.p.  $163^{\circ}$ – $164^{\circ}$  C. This is reduced in acetic acid suspension by hydrogen and platinum black (containing oxygen) to ethyl 1-methylpyrrolidino-2,5-diacetate, b.p.  $162.5^{\circ}$  C. at 9 mm., which after dilution with cymene, is converted by sodium powder at  $172^{\circ}$  C. into ethyl tropinonecarboxylate, from which tropinone is obtained by boiling with 10% sulphuric acid and *r*-p-ecgonine ethyl ester by reduction with sodium amalgam; the latter, after conversion into the methyl ester and benzoylation, yields *r*-cocaine. (Cf. J.C.S., Feb.)—C. S.

**Papain.** V. K. Chesnut. J. Assoc. Off. Agric. Chem., 1920, 3, 387–397.

THE paper deals with the examination of dried papaya latex obtained from papaya trees (*Carica papaya*); the material is easily powdered between the fingers and possesses little or no offensive odour. It is sometimes largely adulterated with starchy substances. A method for determining the activity of the latex depends on its proteolytic action on casein at  $37.5^{\circ}$  C. in solutions having hydrogen ion concentration of  $pH=9.35$ , the action being stopped after 30 mins. by the addition of 30 c.c. of Bogdandy mixture (magnesium sulphate, 50 g., 95% alcohol, 100 c.c., water to 1 l.) and 5 c.c. of *N*/1 hydrochloric acid, the mixture then diluted to a definite volume, filtered, and the filtrate polarised in a 200-mm. tube.—W. P. S.

**Proteins; Precipitation of — by tannins.** T. Solmann. J. Pharm. Exp. Ther., 1920, 16, 49–59.

THE precipitation of proteins by tannin depends on the reaction of the medium. In solutions of the concentration (0.1–0.5%) necessary to produce astringent action precipitation is greatest at  $pH=2-5$ . There is no precipitation when the alkalinity is  $pH>8-8.3$ . The precipitation limits are the same for Witte's peptone as for egg albumin and serum-albumin, and for extracts of catechu as for ordinary tannin. Gallic acid produces, within the same limits, only slight precipitation, probably due to contamination with tannin.—J. C. D.

**Mercury compound; A lipotropic —.** [Determination of mercury.] H. Hüsgen. Biochem. Zeits., 1920, 112, 1–21.

A "LIPOTROPIC" mercury compound, MAT (4-p-toleneazoacetanilide-3-mercurihydroxide,  $CH_3.C_6H_4.N_2.C_6H_4(NH.C_2H_4O).Hg(OH)$ ), is described, which when injected into rabbits is taken up by the central nervous system, the muscles, and other organs much more readily than when ordinary mercury preparations are used. The method used for estimating mercury in the brain consists of the incineration of the organic matter and electrolysis of the mercury, which is afterwards volatilised into a capillary tube which is weighed.—S. S. Z.

**Saccharin and dulcin; Sweetness of —.** T. Paul. Chem.-Zeit., 1921, 45, 38.

THE addition of dulcin to saccharin considerably increases the sweetening power of the latter, although dulcin possesses but little sweetness, e.g., a mixture of saccharin, 280, and dulcin, 120 mg., dissolved in 1 l. of water has the same degree of sweetness as a solution containing 535 mg. of saccharin per l. The taste of the mixture is more pleasant than that of saccharin alone.—W. P. S.

**Hydrazines; Preparation of some —.** L. Thompson. J. Soc. Dyers and Col., 1921, 37, 7–11.

SODIUM hydrosulphite can be used with advantage

in place of bisulphites and stannous salts in the preparation of hydrazine derivatives. Phenylhydrazine, for example, is obtained in more than 90% yield by treating a solution of phenyldiazonium chloride (1 mol.) with a concentrated solution of sodium hydrosulphite (1 mol.) at  $0^{\circ}$  C. in presence of an excess of hydrochloric acid. Complete reduction occurs immediately, and the hydrazinesulphonate is hydrolysed to phenylhydrazine hydrochloride by boiling the solution for a few minutes with a further quantity of concentrated hydrochloric acid. The reactions are expressed by the equations: —  $C_6H_5N_2Cl + Na_2S_2O_4 + H_2O = C_6H_5NH.NHSO_3Na + NaCl + SO_2$ , and  $C_6H_5NH.NHSO_3Na + HCl + H_2O = C_6H_5NH.NH_2 + NaHSO_4$ . Phenylhydrazine-*p*-sulphonic acid is similarly obtained in 60% yield by reduction of diazotised anilphanilic acid with the theoretical quantity of hydrosulphite solution. The reduction of *p*-nitrophenyldiazonium chloride by means of hydrosulphite is best carried out in alkaline solution. The product is a mixture of the sodium salt of *p*-nitrophenylhydrazine and a small quantity of the hydrazinesulphonate. After hydrolysing the latter by boiling with concentrated hydrochloric acid the base is isolated in the usual way, the yield amounting to 95% of the theoretical.—G. F. M.

**Formaldehyde; Preparation of — from ethylene.** R. Willstätter and M. Bommer. Annalen, 1921, 422, 36–46.

By passing a mixture of ethylene (19.38%), oxygen (7.58%), and nitrogen (73.04%) at the rate of 1.5 l. in 13–21 mins. through a Jena glass tube (45 cm. by 3 mm.) heated at  $585^{\circ}$  C., the authors have obtained formaldehyde in 109% (by weight) yield, calculated on the ethylene consumed; that is, about half the quantity theoretically obtainable. The concentration of the formaldehyde in the hot gaseous mixture should not exceed 2% (by vol.)—C. S.

**Hydrogenating metals.** Brochet. See X.

**Lactic acid.** Hofmann. See XVIII.

**Adsorption of alkaloids.** Kolthoff. See XXIII.

**Mercuric chloride and chloroform.** Saase. See XXIII.

#### PATENTS.

**Alkaloid of ergot; Process for the isolation of the principal — in the pure crystallised state.** Chem. Fabr. vorm. Sandoz. E.P. 140,056, 20.12.19. Conv., 10.3.19. Addn. to E.P. 125,396 (J., 1920, 349 A).

THE crude alkaloid obtained by the process described earlier is purified by crystallisation from acetone preferably containing 5–10% of water.—D. F. T.

**Theobromine; Preparation of dialkyl-amino-ethyl derivatives of —.** Soc. Chim. des Usines du Rhône, anc. Gilliard, P. Monnet et Cartier. E.P. 155,748, 27.5.20. Conv., 1.3.20.

DIALKYL-AMINO-ETHYL derivatives of theobromine are obtained by the action of chloroethyl-diethylamines on metallic salts of theobromine. Thus diethylamino-ethyl-theobromine, m.p.  $67^{\circ}$  C., is obtained by boiling for several hours 20 pts. of sodium-theobromine suspended in benzene with 15 pts. of chloroethyl-diethylamine. The base gives a neutral, water-soluble hydrochloride, which after crystallisation from alcohol forms colourless crystals, m.p.  $202^{\circ}$  C. This neutral salt possesses the advantages over other soluble derivatives of theobromine that, whilst having all the therapeutic properties of this substance, it does not excite the mucous membrane and has no irritating effect when used for injection.—G. F. M.

**Acetaldehyde; Process of making** —. H. L. Bender. U.S.P. 1,355,299, 12.10.20. Appl., 20.6.19.  
A mixture of steam and acetylene is heated below 400° C. in the presence of a catalyst.—L. A. C.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Photographic emulsions; Analysis of** —. A. B. Hitchins. Bull. Soc. Franç. Phot., 1920, 7, 228—234.

For the determination of gelatin in photographic emulsion films a Kjeldahl nitrogen estimation is made, and the factor 0.0082 is taken as representing the relation between the number of c.c. of N/10 acid equivalent to the ammonia produced and the weight in grms. of dry gelatin in the sample; air-dried gelatin (as in emulsion films) is taken as containing 13% of moisture. The total silver halide is determined by direct weighing on an asbestos filter, the gelatin being first decomposed and removed with dilute nitric acid. Total silver is determined by Volhard's method, after reducing the halides with zinc and sulphuric acid. Where the halides are only bromide and iodide or bromide and chloride the proportions are obtained from these two figures, graphs being used to simplify the calculation. Where all three halides are present they are detected by converting them into sodium salts and then testing for and removing iodide by persulphate and acetic acid, then testing for and removing bromide by persulphate and sulphuric acid, leaving the chloride still in solution. The iodide can also be quantitatively separated and determined by this method, followed by titration with thiosulphate, the proportion of chloride and bromide in the remainder being determined as outlined above.

—B. V. S.

**[Photographic] reversed dye images; Method of producing** —. J. I. Crabtree. Communication No. 67 from Eastman Kodak Res. Lab. Brit. J. Phot., 1921, 68, 32—33.

If a gelatin film containing a silver image bestained with Methylene Blue and then treated with "acid-hypo" (sodium thiosulphate solution containing free sulphurous acid), the dye in the neighbourhood of the silver image is reduced, but not that in the clear gelatin; by washing the film to remove the leuco-compound thus formed and then dissolving the silver in thiosulphate-ferri-cyanide solution or similar agent a reversed dye image is formed. The silver image becomes much lighter in colour and more transparent, and not completely soluble in the weaker silver solvents such as potassium cyanide. Acid solution of cerous nitrate, acid amidol, and acid stannous chloride act similarly to the "acid-hypo"; a number of other dyes which are easily reduced to leuco-bases behave similarly to Methylene Blue. In practical application of the process it is necessary to select a dye which is removed from gelatin by washing much less easily than its leuco-compound.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

**Nitrocellulose; Method of determining the water-content of wet** —. A. Logothetis and G. Gregoropoulos. Z. ges. Schiess- u. Sprengstoffw., 1921, 16, 1—2.

A known volume of 96% alcohol is stirred with the wet nitrocellulose for 5—10 mins., after which it is filtered, with continued stirring. The volume of the alcoholic filtrate is then carefully measured, and its strength calculated from a specific gravity determination, a correction being applied for contraction in volume.—W. J. W.

**Mercury fulminate; Behaviour of** — with various solvents. A. Langhans. Z. ges. Schiess- u. Sprengstoffw., 1920, 15, 219—221, 227—229, 235—237.

BOTH white and grey fulminate give the pure, white fulminate when their solutions in potassium cyanide are precipitated with nitric acid. The cyanide solutions are not stable. No double salts can be detected in the cyanide solution, nor are these produced by interaction with ammonium nitrate or ammonium sulphate. With ammonium thiocyanate, however, a double salt is formed. A solution of fulminate in 22% ammonia solution (sp. gr. 0.92) yields columnar white crystals of fulminate, which become opaque on treatment with water. The solution decomposes in 12 hrs., and variously coloured sediments are deposited in which no trace of mercuric fulminate is found; these consist of reduction products. A double salt is produced by interaction of the ammonia solution with ammonium thiocyanate. In mono-, di-, and trimethylamine, fulminate dissolves with some difficulty, and with separation of metallic mercury. From the monomethylamine solution white, needle crystals, in fern-shaped masses, are formed; separate needles crystallise out from the di- and trimethylamine solutions. The solutions are not stable. In sodium thiosulphate solution and in aniline solution of the fulminate is accompanied by decomposition. Pyridine dissolves mercury fulminate, and cubical crystals may be obtained, which can be converted into pure fulminate by treatment with water. The pyridine solution is fairly stable, and fulminate precipitated from it after 20 hrs. is of a fair degree of purity, although yellowish in colour. Some separation of mercury, which takes place during solution, is due to interaction of the solvent with organic impurities and not to decomposition of the fulminate. Pyridine as a denaturant for alcohol in the manufacture of fulminate causes low yields, but increases the explosibility of the product. Sodium xanthate dissolves fulminate slightly; the filtrate yields crystals of addition products. On standing decomposition takes place. Mercury fulminate is slightly soluble in acetone and alcohol, but only very slightly soluble in ethyl acetate. It is practically insoluble in benzene, even on heating, and insoluble in chloroform, glycerin, epichlorohydrin, linseed oil, turpentine, and ethyl nitrate. The titration of a solution of fulminate in hydrochloric acid with potassium iodide has been investigated; the mercuric iodide formed first dissolves, but is precipitated on further titration, the line of demarcation being sufficiently definite. Accurate results are obtainable under certain conditions.—W. J. W.

### PATENTS.

**Explosive.** C. Manuelli and L. Bernardini. E.P. 155,627, 17.7.17.

CHLORATES and perchlorates of biguanide and guanidine, prepared by double decomposition of metallic chlorates and perchlorates with salts of biguanide and guanidine, may be used as explosives or constituents of explosive mixtures. Perchlorates of biguanide and guanidine are also obtained by beating a mixture of ammonium perchlorate or chlorate (1 mol.) and dicyanodiamide (2 mols.) to 160°—170° C. The explosives thus prepared, to which oxidising and oxidisable substances may be added, are of good explosive power and possess great stability.—W. J. W.

**Gun-cotton; Process of manufacturing fibrous material, particularly** —. F. A. Wardenburg. Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,355,197, 12.10.20. Appl., 11.12.17.

FIBROUS materials are treated with a liquid in such a manner that the liquid filters through the mass,



and loss of small particles of the material is prevented.—W. J. W.

*Smokeless powders; Manufacture of* —. C. Claessen. G.P. 298,859, 11.3.16.

NITROCELLULOSE, together with nitroglycerin, nitroglycol, or nitrated sugar, is mixed with an eutectic mixture of dinitrotoluene and trinitrotoluene, without the aid of a volatile solvent.—W. J. W.

*Cellulose; Preparation of material from [wood] — which is suitable for nitrating and spinning.* M. Ellern-Eichmann. G.P. 300,703, 19.3.16.

THE treatment of wood cellulose is carried just so far as to give fibres free from incrustation, which are then washed, centrifuged, and dried without the application of pressure or much heat. The product consists of a mass of uncrushed fibres which may be easily separated, and possess good absorptive properties.—W. J. W.

*Gun-cotton; Apparatus for purification of* —. H. Eicheler. G.P. 323,036, 16.9.17.

THE washing vat is provided with two internal sieve plates, into the space between which the gun-cotton is loaded. The wash water enters the bottom of the vat, which is funnel-shaped, and passes upwards through the sieve plates and the material to an overflow at the top. In order to effect complete purification of the smallest particles, the rate of flow of the water is varied periodically, thus ensuring alternate saturation and draining.—W. J. W.

*Delay fuses; Manufacture of* —. F. Israel. G.P. (A) 324,004, 23.1.17, (B) 324,005, 13.7.18, and (C) 324,006, 24.10.18.

(A) NITROCELLULOSE is incorporated with finely-divided charcoal or similar material and an oxygen carrier, such as potassium nitrate, and formed into porous sticks, which are then covered with a layer of an incombustible, or not easily combustible, substance, such as cellulose acetate, and, if desired, enclosed in metal tubes. These fuses are impervious to damp, and burn slowly and uniformly. (B) The nitrocellulose may be used in the form of grains or flakes, pressed into metal or paper tubes. The time of combustion is approximately three times as long as with gunpowder fuses. (C) By omitting the charcoal from the composition, and adding only 1–3% of potassium nitrate, the flakes or grains being then graphitised and pressed into metal or paper tubes, a still slower rate of combustion is obtained.—W. J. W.

*Cellulose and cotton waste; Process and apparatus for drying vegetable materials, especially — before nitration.* Westfälisch-Anhaltische Sprengstoff A.-G. G.P. 326,303, 20.2.18.

THE material is drawn by a fan into a tunnel with heated walls, from which it is then blown direct to the storage chambers in connexion with the nitrating plant.—W. J. W.

*Nitrated material; Process and apparatus for continuous steaming of* —. I. M. Voith. G.P. 326,304, 26.9.18.

THE material is passed continuously through a closed receptacle in which it is subjected to treatment with steam.—W. J. W.

*Damp-proof matches; Process for producing* —. R. Dubrisay. U.S.P. 1,363,095, 21.12.20. Appl., 29.6.20.

As an agglutinant in the paste, a reaction product prepared from an aldehyde and resorcinol, in presence of a catalyst, is employed.—W. J. W.

## XXIII.—ANALYSIS.

*Crucibles used in rock analysis [; Gold-palladium alloy as material for —].* H. S. Washington. J. Wash. Acad. Sci., 1921, 11, 9–13.

APART from the question of cost, a disadvantage in the use of platinum and iridium-platinum crucibles is the difficulty of completely removing the melt after fusion of silicates with sodium carbonate. In experiments with a crucible of "palau" (gold 80%, palladium 20%), the cake obtained after sodium carbonate fusions could be completely and easily removed by treatment with hot water, even when the crucible was indented. Determinations of the loss in weight of crucibles of "palau" and iridium-platinum indicated an average loss, after 47 fusions, of 0.2 mg. with "palau," and with iridium-platinum, after 21 fusions, of 0.5 mg. In the latter case the loss is partly due to volatilisation of iridium.—W. J. W.

*Electric furnace for use in the determination of arsenic by the Marsh method.* L. Birckenbach. Chem.-Zeit., 1921, 45, 61–62.

A SMALL electric furnace for heating the arsenic-mirror tube consists of a kieselguhr brick provided with a groove for holding the tube and fitted with a heating spiral of nickel-chrome wire; the cover of the furnace is also made of kieselguhr, and is divided into two pieces. An alloy of zinc, 500, and copper, 0.625 g. is recommended for use in the hydrogen generation flask in place of ordinary granulated zinc.—W. P. S.

*Indicator; A new* —. R. W. Kinkad. Chem. News, 1921, 122, 4–5.

A NEW indicator of at present unknown constitution, pink in acid, and blue in alkaline media, is formed by the action of ethyl nitrate (1 mol.) on magnesium phenyl bromide (1 mol.) in ethereal solution. The substance is isolated by separating the ethereal layer, evaporating off the ether, adding sodium carbonate and water to the residue, and extracting with chloroform. The blue sodium salt remains in the aqueous layer. It is, however, somewhat unstable, and the indicator is best kept as an ethereal solution of the free acid. Neither the aqueous solution of the free indicator nor that of the sodium salt can be concentrated without decomposition.—G. F. M.

*Acidity in coloured solutions; Titration of* —. B. G. Hartmann. J. Assoc. Off. Agric. Chem., 1920, 3, 410–411.

A MIXTURE of sodium sulphate, 50, and phenolphthalein, 1 g., finely powdered, is recommended for use as external indicator in the titration of strongly coloured acid solutions; drops of the solution are placed on the powder.—W. P. S.

*Adsorption in analytical chemistry. Part. IV. Adsorption of alkalis by cellulose.* I. M. Koltzoff. Pharm. Weekblad, 1921, 58, 46–56. (Cf. J., 1921, 63A.)

WITH sodium and potassium hydroxides no real adsorption occurs, the quantities taken up being proportional to the end concentrations for solutions up to 4N. Between 4N and 6N the quantity taken up is constant, indicating chemical combination; a sudden increase occurs at about 6N, the quantity then remaining constant again. The alkali carbonates are not taken up at all. Barium hydroxide is taken up in amounts proportional to the square roots of the end concentrations. Ammonia is taken up in constant quantity in presence of traces of other alkalis. (Cf. J.C.S., Mar.)—S. I. L.



**Adsorption in analytical chemistry. Part V.** Adsorption of salts of the alkali and alkaline-earth metals and of alkaloids by filter paper. I. M. Kolthoff. Pharm. Weekblad, 1920, 58, 94-101.

PRACTICALLY no adsorption was found for any salts of the alkali or alkaline-earth metals, or for morphine hydrochloride. With quinine hydrochloride and strychnine nitrate small quantities were taken up in exact agreement with the adsorption equation. (Cf. J.C.S., Mar.)—S. I. L.

**Nitrates; Removal of**—[in analysis] by means of alcohol. R. Schneidewind. Chem. and Met. Eng., 1921, 24, 22.

IN analytical processes which require the absence of nitric acid or nitrates, their removal may be more conveniently effected by means of ethyl alcohol than by the usual method of evaporating with sulphuric acid. For example, to a solution containing 20 c.c. of nitric acid and 150 c.c. of water, 15 c.c. of sulphuric acid is added, and when nearly boiling 5 c.c. of ethyl alcohol is carefully run in from time to time, until further addition no longer causes an evolution of nitrous fumes. The boiling is then continued to expel the excess of alcohol and the resulting solution is sufficiently free from nitric acid no longer to give the brown ring test, or to oxidise hydrogen sulphide.—G. F. M.

**Potassium; Separation and determination of**—[as perchlorate]. S. B. Kuzirian. J. Assoc. Off. Agric. Chem., 1920, 3, 321-323.

ANILINE perchlorate is recommended for use in place of perchloric acid; a known weight of the perchlorate is dissolved in 50 c.c. of absolute alcohol, and this solution is added slowly to the mixed chloride solution. For each 1.5 c.c. of water used to dissolve the mixed chlorides, 50 c.c. of absolute alcohol should be added. Sulphates, if present, must be removed previously with barium chloride; the excess of barium chloride does not interfere.—W. P. S.

**Nitrogen; The Kjeldahl method for determining**—I. K. Phelps and H. W. Daudt. J. Assoc. Off. Agric. Chem., 1920, 3, 306-315.

THE method yielded trustworthy results in the case of amines, and pyrrolidine, pyridine, piperidine, quinoline, purine, glyoxaline, quinoxaline, quinoxalene, etc. derivatives, provided that mercuric oxide and potassium sulphate were added to the acid digestion mixture. In the case of azo compounds, a preliminary reduction by boiling with stannous chloride in alcoholic solution was necessary. Hydrazine compounds required boiling with formaldehyde, zinc dust, and hydrochloric acid in alcoholic solution, with subsequent addition of stannous chloride before digestion with sulphuric acid, mercuric oxide, and potassium sulphate.—W. P. S.

**Volumetric analysis. [Determination of mercuric chloride, chloroform, and lead.]** O. Sasse. Pharm. Zeit., 1920, 65, 559, 638.

METHODS are described for the titration of mercuric chloride (with potassium iodide), chloroform (decomposition with potassium hydroxide and estimation of the potassium chloride formed), and lead salts (with potassium bichromate). In the case of mercuric chloride tablets, the red colouring matter which is usually present may be destroyed by treatment with chlorine previous to titration with potassium iodide.—W. P. S.

See also pages (A) 113, *Sulphonic acids* (Van Duin); *Mechanical wood pulp* (Kotihasker). 114, *Reduction of nitrites and nitrates* (Baudisch and Mayer). 115, *Hydrogen cyanide* (Sieverts and

Hermesdorf); *Hydrogen arsenide* (Thoms and Hess). 119, *Zirconium minerals* (Thompson). 121, *Water in oils and fats* (Oertel). 123, *Gelatin and glue* (Ishikawa). 124, *Potash in wood ashes* (Rose); *Ammonia in fertilisers* (Jarrell). 125, *Sugars etc.* (Gilmour); *Lactic acid* (Hofmann); *Milk* (Bouriez, also Ledent). 126, *Beef fat in lard* (Vitoux and Muttelet); *Histidine and arginine* (Kossel and Edlbacher). 127, *Arsenical insecticides* (Roark); *Chinosol* (Spoon). 128, *Papain* (Chesnut); *Determination of mercury* (Hüsgen). 129, *Photographic emulsions* (Hitchins); *Nitrocellulose* (Logothetis and Gregoropoulos).

#### PATENTS.

**Cereals or other substances; Apparatus for indicating moisture in**—Soc. Anon. des Grands Moulins Vilgrain, and M. Chopin. E.P. (A) 143,191, 4.2.20, and (s) 155,488, 25.2.20. Conv., 9.5 and 20.12.19.

(A) THE cereal is introduced in regulated quantity into a heated vertical chamber and then discharged from the same. The amount of water vapour produced is measured by the maximum pressure produced; means are provided for discharging the vapour periodically, and it may be passed to a condenser and the amount of condensed water measured. (s) The cereal is passed downwards through a vertical bronze tube surrounded by an iron tube and provided with an electrical heating jacket. The water vapour from the heated cereal passes through orifices in the bronze tube into the space between the two tubes and escapes thence into a condenser provided with a constant water supply. The difference in temperature of the water entering and leaving the jacket of the condenser is a measure of the amount of water vapour given off by the cereal. The rate of flow of the cereal through the tube is controlled by valves. (Cf. J., 1920, 797 A.)—W. P. S.

#### Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

#### I.—GENERAL; PLANT; MACHINERY.

##### APPLICATIONS.

Arnold, and Goldschmidt A.-G. Carrying out to the limit incompletely-proceeding reactions. 3704. Jan. 31. (Ger., 7.2.20.)

Barbet et Fils et Cie. Evaporating apparatus. 3373. Jan. 27. (Fr., 27.1.20.)

Berk and Co., and Briscoe. Separating solids by crystallisation from solvents. 3848. Feb. 1.

Bibb. Furnace or kiln. 3496. Jan. 28.

Cleworth, Wheal and Co., and Leask. Purification or treatment of air or gas with liquid. 3125. Jan. 25.

Davis. Treating materials in the gaseous phase. 3256. Jan. 26.

Hurrell and Johnson. Rotary filter. 4132. Feb. 4.

Newton. Drying cylinders etc. 4271. Feb. 5.

Paterson. Filtering apparatus etc. 3675. Jan. 31.

Thoresell and Troell. Agglomerating pulverous material. 3965. Feb. 2.

Weyel and Zimmermann. Drying apparatus. 4059. Feb. 3.  
Zack. Obtaining a vacuum by the liquefaction of gases. 4014. Feb. 3.

## COMPLETE SPECIFICATIONS ACCEPTED.

21,323-4 (1918). Francart. Tunnel furnaces, kilns, ovens, etc. (157,474-5.) Feb. 2.  
24,371 (1919). Ruff. Heat treatment or evaporation of liquids. - (157,514.) Feb. 2.  
25,708 (1919). Martinez. Mechanical mixing machines. (157,557.) Feb. 2.  
3894 (1920). Elmore. Centrifugal driers. (158,152.) Feb. 9.  
16,857 (1920). Sharples. Centrifugal machines. (157,688.) Feb. 2.

## II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

## APPLICATIONS.

Accioly. Treatment of mineral etc. oils. 3594. Jan. 29.  
Bibb. Composition for briquettes etc. 3498. Jan. 28.  
Demant. Process for refining hydrocarbons. 4136. Feb. 4.  
Dobson and Mossop. Preparation of liquid fuel. 3634. Jan. 31.  
Fabry. Purifying coal gas. 4007. Feb. 3.  
Farrant. Production of colloidal fuel. 3851. Feb. 1.  
Fletcher. Utilisation of peat etc. for steam production. 3114. Jan. 25.  
Halbergerhütte. 3238. See X.  
Illingworth. Coking coal. 3923. Feb. 2.  
Kneen. Water-gas plant. 3109. Jan. 25.  
Maus. Drying and pressing peat into shape. 3823. Feb. 1.  
Torfverwertungs Ges. Pohl u. von Dewitz. Dry distillation and coking of raw peat etc. 2978. Jan. 24. (Ger., 22.1.20.)  
Trent Process Corp. Production of coke. 4187. Feb. 4. (U.S., 21.2.20.)  
Wilton. Utilising waste gases from furnaces etc. for extracting by-products from coal etc. 3407. Jan. 28.

## COMPLETE SPECIFICATIONS ACCEPTED.

12,509 (1918). Garrow. Low and medium temperature carbonisation of coal, shale, wood, peat, etc. (158,002.) Feb. 9.  
18,038 (1919). Broadbridge and others. See XII.  
4591 (1920). Melamid. Manufacture of transformer oil. (143,193.) Feb. 9.  
17,763 (1920). Miles. Manufacture of paraffin wax emulsions. (145,602.) Feb. 9.

## III.—TAR AND TAR PRODUCTS.

## APPLICATIONS.

Bawden, Bennett, and United Alkali Co. Manufacture of *o*- and *p*-nitrophenol. 3369. Jan. 27.  
Bawden, Bennett, and United Alkali Co. Separation of *o*- and *p*-nitrochlorobenzenes from mixtures thereof. 3370. Jan. 27.

Demant. 4136. See II.

Prager, and South Metropolitan Gas Co. Manufacture of naphthalene compounds. 3032. Jan. 24.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

## APPLICATIONS.

Addy, and British Cellulose and Chem. Manuf. Co. Manufacture or treatment of articles made of plastic materials. 3902. Feb. 2.  
Bernot and Fournier. Manufacture of paper pulp. 3828. Feb. 1.  
Bloxiham (Akt.-Ges. f. Anilinfabr.). Protecting animal fibres in treating them with alkaline liquids. 3377. Jan. 27.  
Cew and Marx. 3898. See XIII.  
Duclaux. Treatment of cellulose esters. 3742. Feb. 1.  
Joliot. Manufacture of cellulose threads. 4191. Feb. 4. (Fr., 30.8.20.)  
Schwartz. Manufacture of cellulose acetate and artificial silk therefrom. 3296. Jan. 27.  
Sutcliffe. Preparation of vulcanised fibre. 3920. Jan. 27.

## COMPLETE SPECIFICATIONS ACCEPTED.

21,172 (1919). Goldschmidt. Process of degumming silk. (131,906.) Feb. 2.  
16,075 (1920). Glanzfäden A.-G. Preparing durable cupric ammonia cellulose solutions for spinning artificial thread etc. (145,035.) Feb. 9.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

## APPLICATIONS.

Bloxiham (Akt.-Ges. f. Anilinfabr.). 3377. See V.  
Briggs, Richardson, and British Cellulose and Chem. Manuf. Co. Dyeing. 3347. Jan. 27.  
Calico Printers' Assoc., and Costobadie. Production of ornamental effects on fabrics. 3486. Jan. 28.  
Calico Printers' Assoc., and Nelson. Ornamentation of textile fabrics. 4073. Feb. 9.  
Tate. Process for dyeing and waterproofing. 3198. Jan. 28.  
Whitaker and Whitaker. Machines for dyeing, scouring, and washing wool etc. 3172. Jan. 28.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

## APPLICATIONS.

Barnett. Production of chemical compounds or elements by gravitational translation of amalgam formed by electrolysis to hermetically sealed receptacle. 3171. Jan. 28.  
Freeth and Munro. Production of ammonium chloride and sodium carbonate. 2921. Jan. 24.  
Nicolli. Production of hydrochloric acid by direct synthesis. 3104. Jan. 25. (Ital., 2.3.20.)  
Parrish, and South Metropolitan Gas Co. Manufacture of ammonium sulphate. 3033. Jan. 24.  
Soc. l'Air Liquide. Synthesis of ammonia. 3949. Feb. 2. (Fr., 2.2.20.)

Wade (Lindsay Light Co.). Manufacture of thorium compounds. 4100. Feb. 3.

#### COMPLETE SPECIFICATIONS ACCEPTED.

25,544 (1919). Mackay. Making lead sulphate direct from lead sulphide ores. (157,554.) Feb. 2.  
26,332 (1919). Armour Fertilizer Works. Production of aluminium nitride. (146,919.) Feb. 9.  
10,761 (1920). Soc. l'Air Liquide. Production of hydrogen peroxide. (141,758.) Feb. 9.

### VIII.—GLASS; CERAMICS.

#### APPLICATIONS.

Ferguson. Manufacture of sheet glass. 3144. Jan. 25.  
Marlow. Ovens or kilns for manufacture of tiles, pottery, etc. 3526, 3580-1. Jan. 29.

#### COMPLETE SPECIFICATIONS ACCEPTED.

27,438 (1919). Frink. Manufacture of sheet glass. (157,598.) Feb. 2.  
16,191 (1920). Truel. Glass-melting tanks or furnaces. (157,684.) Feb. 2.

### IX.—BUILDING MATERIALS.

#### APPLICATIONS.

Lyon. Process for drying wood. 3950. Feb. 2.  
Marks. Concrete. 3975. Feb. 3.  
Newberry. Manufacture of cement. 3707. Jan. 31.  
Winkler. Manufacture of mortar, cement, etc. 3783. Feb. 1. (Switz., 26.7.20.)  
Winkler. Cement, mortar, concrete, etc. 3903-4. Feb. 2. (Switz., 30.8 and 15.10.20.)

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

#### APPLICATIONS.

Alexander. Nickel alloy. 3306. Jan. 27.  
British Thomson-Houston Co. (General Electric Co.). Alloys. 4088. Feb. 3.  
Fairweather (Avesta Jernverks Akt.). Producing silicon-manganese-chrome steel. 3672. Jan. 31.  
Fehr and Fehr. Electrodeposition of bronze etc. 3097. Jan. 25.  
Fletcher. Smelting, melting, and refining metal in furnaces using air blast. 3108. Jan. 25.  
Freedman and Greetham. Extraction of metals from their compounds. 3159. Jan. 26.  
Goldschmidt A.-G. Bearing metal alloy of high lead content. 3251. Jan. 26. (Ger., 26.1.20.)  
Gunderson. Process of case-hardening copper. 3715. Jan. 31.  
Halberghütte Ges. Purification of blast-furnace etc. gases. 3238. Jan. 26. (Ger., 23.3.20.)  
Hale. Production of malleable iron castings. 4072. Feb. 3.  
Iytaka. Alloy. 3487. Jan. 28.  
Lowe. Soldering aluminium. 2918. Jan. 24.  
Pacz. Alloys, and process of treating same. 3260. Jan. 26. (U.S., 13.2.20.)

Rheiniach-Nassauische Bergwerks- u. Hütten A.-G., and Spicker. Production of zinc dust having a high percentage of metallic zinc. 3481. Jan. 28. (Ger., 22.11.20.)

Rosthorn. Manufacture of copper alloys. 4230. Feb. 4. (Ger., 4.2.20.)

Trent Process Corp. Treating ore etc. 4188. Feb. 4. (U.S., 21.2.20.)

Trent Process Corp. Collecting and purifying minerals. 4189. Feb. 4. (U.S., 9.4.20.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,909 (1919). Armstrong. Stable surface alloy steel. (143,829.) Feb. 2.

18,498 (1919). Thompson, Ltd., Thompson, and Partridge. Coating iron, steel, and certain other metals with aluminium. (158,010.) Feb. 9.

25,544 (1919). Mackay. See VII.

25,546 (1919). Mackay. Recovery of vanadium from its ores. (157,555.) Feb. 2.

32,093 (1919). Schafer and Brettschneider. Case-hardening mixtures for iron and steel. (157,639.) Feb. 2.

5722 (1920). Hall, and Rolls-Royce, Ltd. Preparation of alloys and refining metals and alloys. (158,156.) Feb. 9.

24,670 (1920). Russell, and Manchester Furnaces, Ltd. Heat-treatment furnaces. (157,606.) Feb. 2.

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Automatic Telephone Manuf. Co., Ltd., and Roseby. Electric furnaces. 4247. Feb. 5.

Barnett. 3171. See VII.

Fehr and Fehr. 3097. See X.

Pouchain. Negative electrode for electric accumulators. 3378. Jan. 27.

#### COMPLETE SPECIFICATION ACCEPTED.

32,790 (1919). Levin. Electrolytic gas generators. (158,148.) Feb. 9.

### XII.—FATS; OILS; WAXES.

#### APPLICATIONS.

Bitterling, Lyman, and Bitterlings, Ltd. Apparatus for rendering fats, drying, sterilising, or destroying animal matter. 3586. Jan. 29.

Fankhauser. Squeezing oils and fats from oily and fatty substances. 3922. Feb. 2. (Switz., 3.2.20.)

Green. 3741. See XIX.

Tempelhoff. Medicated antiseptic soap. 2946. Jan. 24.

#### COMPLETE SPECIFICATIONS ACCEPTED.

18,038 and 19,797 (1919). Broadbridge, Edser, and Minerals Separation, Ltd. Treatment of emulsions and the like. (157,490.) Feb. 2.

6239 (1920). Boehringer Sohn. Manufacture of soaps and washing-materials. (139,776.) Feb. 2.

13,582 (1920). Wade (Wilson and Co.). Process for compounding fats and oils. (158,175.) Feb. 9.

15,882 (1920). Elektro-Osmose A.-G. Process for decolorising liquids containing glycerin or crude glycerin. (144,727.) Feb. 2.

**XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.****APPLICATIONS.**

Cew and Marx. Preparation of dilute solutions of resin soap. 3898. Feb. 2.

**XIV.—INDIA-RUBBER; GUTTA-PERCHA.****APPLICATIONS.**

Davidson. Treatment of rubber etc. 4306. Feb. 5.

**XV.—LEATHER; BONE; HORN; GLUE.****APPLICATIONS.**

Hayward. Apparatus for drying gelatin etc. 3896. Feb. 2.  
Schidrowitz. Adhesives. 3388. Jan. 27.

**COMPLETE SPECIFICATIONS ACCEPTED.**

16,743 (1920). McQuitty. Manufacture of sheets of edible gelatin. (145,486.) Feb. 9.  
19,827 (1920). Devoz. See XVI.

**XVI.—SOILS; FERTILISERS.****COMPLETE SPECIFICATIONS ACCEPTED.**

17,608 and 17,720 (1920). Akt.-Ges. f. Anilinfabr. Manufacture of artificial fertilisers. (146,259 and 145,582.) Feb. 9.  
19,827 (1920). Devoz. Manufacture of soluble nitrogenous manures from waste leather and other animal detritus. (147,798.) Feb. 9.

**XIX.—FOODS; WATER PURIFICATION; SANITATION.****APPLICATIONS.**

Bitterling and others. 3596. See XII.  
Dick, and International Dry-Milk Co. Dehydrated milk. 4208. Feb. 4.

Green. Purification and deodorisation of sewage oil or grease. 3741. Feb. 1.

Smith. Concentrated coconut milk. 4074. Feb. 3.

Smith. Coconut food products. 4075. Feb. 3.

Trent. Sewage process. 3420. Jan. 28.

**COMPLETE SPECIFICATIONS ACCEPTED.**

24,915 (1919). McDougall and Howles. Sheep and cattle dips and washes. (157,527.) Feb. 2.  
26,785 and 31,759 (1919). Brock. Baking-powder or self-raising flour. (157,581.) Feb. 2.

**XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.****APPLICATIONS.**

Chem. Fabr. auf Actien, vorm. E. Schering. Manufacture of diethylbarbituric acid compound. 3138. Jan. 25. (Ger., 261,20.)  
Douse, and United Alkali Co. Manufacture of ethyl chloroformic ester. 3371. Jan. 27.  
Metcalf and Usher. Extracting essential oils etc. 3995. Feb. 3.

**COMPLETE SPECIFICATION ACCEPTED.**

26,700 (1919). Sidgwick, Plant, and Boake, Roberts, and Co. Preparation of diethyl sulphate. (157,578.) Feb. 2.

**XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.****APPLICATIONS.**

Brandenberger. Production of sensitive photographic films. 3252. Jan. 26.  
Brandenberger. Photographic films with a carrier permeable to water. 3253. Jan. 26.  
Soc. Anon. La Cellophane. Preparation of photographic films permeable to water. 2981. Jan. 24. (Fr., 19,420.)  
Sperati. Photography. 3817-8. Feb. 1. (Ital., 17 and 16,10,20.)

**COMPLETE SPECIFICATION ACCEPTED.**

23,910 (1919). Donisthorpe, and Dye Impression Photos, Ltd. Processes of dye transfer printing from photographic negatives. (158,021.) Feb. 9.

**I.—GENERAL; PLANT; MACHINERY.**

*Soot and sulphur compounds; Formation of — in boiler plants.* E. Donath. Brennstoff-Chem., 1921, 2, 26—28.

THE chemical character of soot and of the sulphur compounds of flue gases is discussed, also their corrosive effects on ironwork in the cooler parts of the plant. These are most formidable in the products from such fuels as the Arsa coal of Istria, which contains 10% of sulphur. When thrown upon a hot fire this evolves at first large volumes of hydrogen sulphide, which attacks even the iron of the fire doors with the formation of ferrous sulphide. The flue dust contains sulphides, and is also corrosive. Some advantage is gained by admixture of lime and bog iron ore, but it is concluded that such coal is unsuitable for boiler firing. As the coal gives a large yield of low-temperature tar in a suitable gas producer, its gasification is suggested, with recovery of sulphur by oxide purification, prior to using the gas as fuel.—H. J. H.

**PATENTS.**

*Pulveriser and process of fine pulverising.* F. Seymour, Assr. to Aero Pulveriser Co. U.S.P. 1,363,361, 28.12.20. Appl., 7.1.19.

A NUMBER of rotary beaters are arranged on a horizontal shaft which also carries a fan at one end, and the whole is enclosed in a casing. The beaters decrease in diameter from the fan towards the other end of the series, and the casing is correspondingly stepped. Annular partitions project inwards from the periphery of the casing between the beaters, and the material is carried horizontally through the apparatus by the air current.—W. F. F.

*Ball mill.* H. Sellman. U.S.P. 1,363,620, 28.12.20. Appl., 26.2.20.

A HORIZONTAL pulveriser drum is provided with an outlet disc valve on its periphery, having the valve stem projecting outwards through the casing. A space is provided in the wall of the drum between the valve disc and the outer surface of the casing, and the valve spindle is packed where it passes through the latter. The valve disc is cut into sectors and moves over a plate perforated in similar sectors which controls the passage from the drum to the space within the wall.—W. F. F.

*Orre-grinding machine.* G. W. Morthland. U.S.P. 1,363,990, 28.12.20. Appl., 20.12.18.

MATERIAL is admitted through a hollow trunnion to a horizontal grinding drum which gradually increases in diameter towards the outlet end, and the ground material is discharged through the other trunnion. The drum contains a number of transverse discs of equal diameter, but slightly smaller than the smallest diameter of the drum, and increasing in thickness towards the discharge end of the drum. Each disc is provided with a central opening so as to form a continuous passageway through the apparatus, and with radial grooves in their meeting faces. The discs are packed in the drum loosely, so that on rotation they are free to rotate relatively to one another.—W. F. F.

*Electro-osmotic separation of substances.* Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 144,710, 10.6.20. Conv., 8.4.18.

In the electro-osmotic separation of substances consisting of dispersoids, such as suspension or emulsion colloids, ions and non-ionised substances, ions suitable for the purpose of electro-osmosis are substituted for ions unsuitable for this purpose in the mixture under treatment. In the purification of albumin solutions containing ammonium sulphate,

for example, the ammonium ions migrate more quickly than the  $\text{SO}_4$ -ions, with the result that the solution in the middle compartment of the electro-osmotic cell becomes acid and albumin is precipitated. In such case the  $\text{SO}_4$ -ion is preferably replaced by the anion of acetic acid by addition of barium acetate and removal of the precipitated barium sulphate. In like manner univalent ions can be exchanged for other univalent ions, and multivalent anions replaced by suitable organic or inorganic anions of the same or different valency.—J. S. G. T.

*Metal [heat] regenerators.* M. Mathy. E.P. 147,035, 6.7.20. Conv., 4.6.18.

THE regenerator is constructed of metal tubes of small diameter supported on slabs or arches of refractory material to prevent buckling.—B. M. V.

*Furnace reaction chamber and method of preventing destruction thereof.* F. J. Metzger, Assr. to Air Reduction Co., Inc. U.S.P. 1,363,428, 28.12.20. Appl., 6.2.20.

NICHROME tubes which are used in carrying out high temperature reactions by external heating with oxidising gases are protected by an internal coating of nickel.—W. F. F.

*Annealing oven.* S. J. Booth, J. Terreault, and W. L. Burn. U.S.P. 1,364,694, 4.1.21. Appl., 20.6.19.

A BRICKWORK chamber is divided by a partition into a firebox compartment and an oven compartment. The walls, roof, etc., of the latter are double, the spaces between forming flues through which the burning gases from the firebox are led as follows:—Along the roof, down the end wall, along the bottom, up the partition wall, along the top part of the side walls, back along the bottom part of the side walls to the exhaust flue.—B. M. V.

*Salts; Means for drying —.* South Metropolitan Gas Co., and P. Parrish. E.P. 156,963, 14.11.19.

In a drying apparatus of the kind in which the salt falls in a shower down a tower through which an upward current of hot gases is maintained, the tower is provided with distributing cones at different levels; baffle bars of triangular cross-section are disposed between the cones; devices are provided for directing on to the next cone the salt which has been distributed by the preceding one; and the surfaces which receive the descending salt are subjected to continuous or intermittent vibration.—W. E. F. P.

*Drying apparatus.* W. H. Perry, Assr. to Normandy Sea Food Co. U.S.P. 1,363,431, 28.12.20. Appl., 26.12.18.

A CONDUIT through which a conveyor passes is arranged just above another conduit through which warm air is forced. A number of communicating passages are provided so that regulated quantities of warm air may be admitted into the conveyor conduit and through the conveyor.—W. F. F.

*Drying liquids and the like; Method of —.* P. Müller. U.S.P. 1,364,403, 4.1.21. Appl., 26.8.13. Renewed 28.5.20.

LIQUID is finely subdivided and projected horizontally across a drying chamber in a flat lamina. A drying medium is also projected horizontally, in the same direction, over the top and bottom surfaces of the lamina, and is maintained in contact therewith until the particles are dried.—W. F. F.

*Drying apparatus.* O. Müller. U.S.P. 1,364,402, 4.1.21. Appl., 25.8.20.

A DRYING apparatus comprises a number of trucks

placed end to end and divided by horizontal partitions so as to form several superposed continuous conduits. Hot air is blown at one end into all the conduits except the uppermost, and the air passes at the other end into the uppermost conduit, through which it passes back to the outlet.

—W. F. F.

*Heater [for obtaining hot air with reduced oxygen content].* J. McC. Selden, Assr. to The Selden Co. U.S.P. 1,352,481, 14.9.20. Appl., 12.2.18.

THE heater consists of a chamber with a haffle wall in the middle, this wall being vertical on one side and stepped on the other. A flame is projected into the chamber against the lower broader end of the stepped side of the haffle wall. Air passed through the chamber, upwards over the stepped side of the haffle wall and then downwards over the other side, can be rapidly heated to any temperature from 150° to 1000° C., and at the same time its oxygen content can be reduced to 14%, thus rendering it suitable for use in the catalytic oxidation of naphthalene to phthalic anhydride (cf. E.P. 119,517—8; J., 1918, 684 A) without the risk of producing readily explosive mixtures.

*Emulsifier.* C. B. Dalzell, Assr. to D. H. Burrell and Co. U.S.P. 1,363,572, 28.12.20. Appl., 24.11.19.

A CYLINDRICAL vessel is carried at one end of a horizontal shaft, and liquid is admitted axially into its open end from a fixed tank. The liquid is discharged over the edge by centrifugal force into a second annular vessel, and from that vessel into the surrounding casing. The shaft is packed so as to prevent the admission of air into the casing.

—W. F. F.

*Gases; Apparatus for bringing about and controlling reactions between —.* C. Conover. U.S.P. 1,363,955, 23.12.20. Appl., 30.4.19.

A REACTION chamber for gases consists of a sinuous pipe within a casing. The casing is provided with partitions which extend from each side wall alternately and cause air which is passed through the chamber to follow the bends of the pipe.—W. F. F.

*Gases; Process of and apparatus for treating mixed —.* C. S. Palmer. U.S.P. 1,364,136, 4.1.21. Appl., 28.10.18.

A MIXTURE of two gases, one of which is more susceptible to magnetisation than the other, is passed over and through a porous magnetised screen. The gases are withdrawn in two streams at opposite sides of the screen, one of the streams being enriched with the more magnetisable gas.—W. F. F.

*Mixing apparatus.* R. W. Shafor, Assr. to The Dorr Co. U.S.P. 1,364,412, 4.1.21. Appl., 26.7.16.

AN apparatus for mixing liquid with a gas comprises a closed container having a lateral outlet for the treated liquid, and a vertical conduit extending from the lower end to a point above the liquid level. The gas is forced under pressure into the lower end of the conduit so as to form a gas-lift. Excess gas is returned from the gas space above the liquid and returned to the gas-supply pipe between the source of supply and the compressor.

*Pulverulent material; Means for preparing and conveying —.* W. O. Amsler. U.S.P. 1,364,603, 4.1.21. Appl., 3.4.16.

MATERIAL is pulverised between crushing rolls and a horizontal table which is heated so as to dry the material. The material is withdrawn by means of a pipe which is moved over the surface of the table. Suction is applied to the pipe so as to draw preheated air into the casing and thence to the open end of the pipe.—W. F. F.

*Extracts; Apparatus for making —.* T. P. Tuite. U.S.P. 1,365,068, 11.1.21. Appl., 8.5.17.

A RESERVOIR for the extracting medium communicates with a container for pulverised material through an opening that can be adjusted by changing the position of the reservoir.—B. M. V.

*Photochemical apparatus.* W. O. Snelling. U.S.P. 1,365,740, 18.1.21. Appl., 22.11.16.

THE apparatus consists of a series of transparent vessels, each having an inlet and an outlet, and provided with screens which are adjustable independently.—W. E. F. P.

*Separating the constituents of air or other gaseous mixtures; Apparatus and method for —.* R. Wucherer and F. Pollitzer, Assrs. to The Linde Air Products Co. U.S.P. 1,360,853, 30.11.20. Appl., 29.12.14.

SEE E.P. 24,735 of 1914; J., 1915, 871.

*Kiln.* F. Fidler and J. G. Maxwell. U.S.P. 1,364,090, 4.1.21. Appl., 27.12.19.

SEE E.P. 141,124 of 1919; J., 1920, 408 A.

*Rotary apparatus for treating loose material with gaseous fluids.* A. Gerlach. U.S.P. 1,364,105, 4.1.21. Appl., 6.3.20.

SEE E.P. 133,327 of 1919; J., 1920, 143 A.

*Filtering or dewatering press.* A. ten Bosch, Assr. to Naaml. Vennoots. Nederlandsche Veenverwerking Maatschappij. U.S.P. 1,366,307, 18.1.21. Appl., 24.4.20.

SEE E.P. 154,817 of 1920; J., 1921, 71 A.

*Crushing and pulverising machines.* J. E. Kennedy. E.P. 139,219, 20.2.20. Conv., 2.2.14.

*Casings for glass containers.* E.P. 131,918. See V.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Mineral matter in coal; Distribution of —.* R. Lessing. Inst. Min. Eng., Jan. 22, 1921. [Advance proof.]

SAMPLES of colliery slack were subjected to elutriation with an upward current of water; the dust was first removed from the surface of the slack by the action of a slow current, and the coal subsequently freed from the "dirt" by increasing the speed of the current, the coal overflowing and the dirt remaining behind. The ash content of the dust thus collected was generally about 20%, whilst that of the coal varied from 1.44 to 1.98% and that of the dirt from 23.95% to 54.69%. The ash contributed to the entire sample by the dust was at least equal to, and in some cases more than twice as much as that contributed by the whole bulk of the clean coal, although the dust portion itself only ranged from 6 to 9% of the bulk of the sample. The ash was separated into water-soluble, acid-soluble, and acid-insoluble portions, and each of these portions was separately examined. In a Staffordshire coking slack the water-soluble portion of the ash from the dust amounted to 2.44%, of the ash from the clean coal to 3.48%, and of the ash from the dirt to 3.70%; the acid-soluble portion of the ash from the dust was 23.72%, of that from the clean coal, 50.50%, and of that from the dirt, 38.96%, whilst the acid-insoluble portion of the ash from the dust amounted to 73.84%, of the ash from the clean coal to 48.02%, and of the ash from the dirt to 57.34%. The ash in light clean coal decreases with the size down to 30-mesh, with an increase in the portion through 30-mesh, the latter doubtless due to its containing fusain dust; whilst that of the durain

portion shows a decrease down to 20-mesh, followed by an increase in the smaller size. The ash in the dirt consistently increases with decreasing size. In the float-and-sink method of separating coal and "dirt," the author uses as liquid carbon tetrachloride or a mixture of this with light petroleum spirit.—A. G.

*Coal examination; A simplified method of —.* M. Dolch. Mitt. Inst. Kohlenvergasung, 1921, 3, 1—6.

THE dried coal is heated in a transparent quartz tube, inserted in an electric furnace wound with nichrome wire, and lagged with kieselguhr, this being retained in place by asbestos cord, painted over with sodium silicate. The determinations of coke, moisture, ash, tar yield, and quality and quantity of gas are all made in one apparatus. To avoid partial combustion the carbonisation is carried out in an atmosphere of nitrogen or carbon dioxide. The passage of a current of dry air over the tar in a special apparatus greatly accelerates the drying of the tar. The production of tar fog is avoided by slow heating and the use of dried coal. Moisture is estimated by attaching a tube containing calcium chloride saturated with carbon dioxide and weighing before and after a period of one hour during which time the temperature is maintained at 105° C. and a current of carbon dioxide is passed through the tube. Coke is estimated by carbonisation at 800° C. for half an hour, the carbonisation tube being weighed before and after the experiment. The weight of tar plus gas liquor can be obtained from the weight of the tar tube, and by subsequent drying the percentages of water of constitution and anhydrous tar are obtained.—A. G.

*Carbon dioxide in coal; Determination of —.* F. S. Sinnatt and W. Harrison. Bull 7, Lancs. and Ches. Coal Research Assoc., 1920, 15 pp.

THE sample of coal is pulverised to pass through a 1/90-mesh sieve, and from 0.5 to 5 g. is introduced into the decomposition flask of the apparatus described previously (Analyst, Apr., 1913). The coal is covered with about twice its volume (5 to 15 c.c.) of distilled water, and is then gently heated, the contents being open to the air, until the water commences to boil, and maintained at a gentle simmer for about 20 mins. The carbonates in the coal are decomposed with hydrochloric acid (5%) or phosphoric acid (syrupy acid diluted 4 times), and the carbon dioxide is carried over by a constant current of air into a collecting vessel, where it is estimated by Pettenkofer's method.—W. P.

*Coke ovens; Pyrogenic decompositions in —.* A. Thau. Brennstoff-Chem., 1920, 1, 52—53, 66—68.

CARBONISATION trials with laboratory and small-scale apparatus are not likely to furnish the same results as large-scale plant, particularly as regards the secondary pyrogenic reactions. The author has therefore made observations on a single coke oven in a working battery carbonising Durham coal. The products from this were isolated from the rest, the crude gas being drawn through a small cooling and washing plant consisting of a train of oil casks and then collected for analysis in a small holder. The temperature of the oven wall at charging was varied and measured, as also the temperature attained at the oven arch. After determining the nature of the products at a certain temperature with the oven carefully sealed, a quantity of air (at the higher temperatures, oxygen from cylinders) was drawn in above the charge so as to increase the temperature of the arch by 100°—160° C. Oxygen was used to avoid the disturbing influence of nitrogen on the results. The nature of the crude gas and products then drawn from the oven was again determined. In this way the effect of in-

drawn air due to "overpulling" could be determined under various conditions of carbonisation. The "low temperature" character of the products of carbonisation at 500° C. became less marked as the initial wall temperatures were raised. The effect of in-drawn air at 500°—800° was not serious; it acted merely as a diluent. Above 900° the products were essentially of a "high temperature" character. The crude benzol showed traces only of paraffins and the gas contained traces of naphthalene. No serious decomposition of ammonia was apparent even at 1000° in the absence of oxygen, but the naphthalene increased at the expense of the benzol. By an experiment on a charge of three-quarters the normal size it was shown that the effect of an empty space was less than that due to contact with glowing carbon. In general the decompositions were not serious even at the highest temperature until oxygen was admitted. Then a rapid loss of ammonia was observed, naphthalene was formed at the expense of the benzol, and the tar became thicker. The author concludes that the induction of air during carbonisation is the main cause of degradation of products, and therefore that a negative pressure in gas retorts and coke ovens is to be avoided as far as possible.

—H. J. H.

*Air factor; Calculation of the — [during combustion].* A. B. Helbig. Feuerungstech., 1921, 9, 53—58.

A METHOD is worked out for the calculation of the air requirements of liquid and gaseous fuels from an analysis of the flue gases with the aid of a triangular diagram (cf. Ostwald, J., 1919, 492 A). —W. P.

*Gas producers; Use of steam for cooling the fuel bed of —.* H. Koschmieder. Brennstoff-Chem., 1921, 2, 23—26.

IN a previous communication (cf. J., 1921, 111 A) it was shown that 786 kg.-cals. of heat must be absorbed per 1 kg. of carbon gasified to reduce the temperature of the bed to 1000° C. when cold air blast is used. This may be done by the injection of steam which is effective either by the absorption of heat in water-gas production or by increase of the sensible heat of the steam. The necessary heat would be absorbed by 0.409 kg. of steam if completely decomposed, or by 1.974 kg. if no decomposition occurred. The author has worked out and tabulated the effects of additions of steam between these limits on the quantity, composition, and calorific value of the gas, and the efficiency of its production, both when the air blast is cold and heated to 500° C. He has further calculated and tabulated the results of such additions on the efficiency of the gases in use when burnt under different conditions, e.g., with and without recuperation.—H. J. H.

*Wood and wood waste; Gasification of — in producers.* Gwosdz. Brennstoff-Chem., 1920, 2, 21—23.

IT is necessary to remove moisture from wood gas to secure high flame temperatures, and tarry matter if the wood gas is to be used in gas engines. The latter is specially important, and the methods employed in gas producers of British, French, and American types are described briefly. Two producer plants of German construction are described in more detail. One consists of a producer, into which air is blown below a horizontal grate, and fitted with a bell and charging hopper. Cooling and partial separation of tar and dust are effected in a vertical tower fitted with water spray. Purification is completed in a centrifugal gas cleaner followed by an impact tar separator. The charge of wood waste must be well mixed if the degree of coarseness varies, and a proportion of fines is advantageous.



Another producer has superposed a tall distillation shaft and is capable of dealing with wood waste above 20 mm. size if containing not more than 25% of water.—H. J. H.

*Petrol; Blended* — containing casinghead gasoline. F. Bordas. Ann. Falsif., 1920, 13, 539—543.

In view of recent importations into France of blended gasoline, investigations have been instituted by the Ministry of Finance to differentiate between these spirits and straight run petrol, in view of the increased fire risks and the marked heavy residue. Modifications of the current tests are described.—A. E. D.

*Mineral oils; Estimation of pitchy substances (asphalt and resin) in* —. G. Armani and A. G. Rodano. Giorn. Chim. Ind. Applic., 1920, 1, 45—48.

THE usual method of determining asphaltic and pitchy substances in mineral oil by shaking a light petroleum solution of the oil with concentrated sulphuric acid, allowing to settle, measuring the volume of the dark lower layer and subtracting therefrom the volume of the sulphuric acid used (cf. Villavecchia, "Applied Analytical Chemistry," Vol. I, p. 339), occasionally fails, the volume of the pitchy matter being very high and sometimes exceeding that of the oil employed. In such cases the following method may be employed: A weighed quantity of the oil (about 10 c.c.) is dissolved in a separating funnel, in 100 c.c. of light petroleum (sp. gr. not above 0.700), which should not be attacked by concentrated sulphuric acid; 5 c.c. of concentrated sulphuric acid (66° B., sp. gr. 1.84) is added and the funnel stoppered and shaken energetically for some minutes and then left at rest for at least 12 hrs. When the two layers have separated, 50 c.c. of the clear petroleum layer is pipetted off, washed with water in another separating funnel until neutral and placed in a tared flask. The light petroleum is then distilled off, and the residue dried at 100° C. and weighed. Multiplication of this weight by 2 gives the amount of pure mineral oil in the amount used. With oils containing normal proportions of pitchy substances the results obtained by this new method agree with those given by the old method, but when the latter yields a volume of pitchy matter equalling or surpassing that of the oil itself the authors' procedure yields much lower results. The new method is applicable to mineral lubricating oils and also to the distillation residues of mineral oils. With mineral oils containing fatty substances or blown vegetable oils it is necessary first to remove the fatty substances by saponification.—T. H. P.

*Lubricating oils; Methods of examination of* —. G. F. Robertshaw. J. Inst. Petrol. Techn., 1920, 6, 324—364.

PHYSICAL and chemical properties of lubricating oils are discussed in some detail, and published methods of testing them are described briefly. A bibliography is appended.—A. E. D.

*Lubricating oils in internal combustion engines; Carbonisation of* —. F. H. Garner. Inst. Petrol. Techn., 18.1.21. [Advance proof.]

ASPHALTIC resins in lubricating oils may be estimated by means of the adsorptive power exhibited by animal charcoal. A known mass of the oil (20 g.) mixed with 100 g. of charcoal is extracted in a Soxhlet apparatus with petroleum ether for 2 hrs., and the recovered oil is weighed. In similar oils the resin content influences both the extent of carbonisation and the Conradson coke value (J., 1912, 912; Amer. Soc. Testing Materials Standards, 1918, 620). Oils of similar distillation range from Texas and Pennsylvania crude oils

show similar evaporation losses at the same temperature of evaporation, and there seems to be a direct relation between the evaporation loss and fire test. As a criterion of the behaviour of an oil in the engine, with regard to formation of carbon deposits, the Conradson coke value is superior to the carbonisation value, i.e., the percentage of asphaltenes formed on heating for a definite period at a fixed temperature. The subjoined table gives some characteristic results:—

Oil.	Heated for 4 hrs. at	Resin content.	% asphaltenes formed.	Conradson coke value.
Light distillate ..	200° C.	1.2	1.36	0.19
" " (refined) ..	200° C.	0.6	0.50	—
Heavy distillate ..	200° C.	2.4	0.60	0.075
" " (refined) ..	200° C.	1.2	0.40	—
Cylinder stock ..	300° C.	5.11	2.27	2.6

A very full bibliography appertaining to the various tests for lubricating oils is given.—A. E. D.

*Montan wax from lignite; Extraction of* —. E. Donath. Brennstoff-Chem., 1920, 1, 86—87; 1921, 2, 28.

ACETONE is suggested as a substitute for the benzol-alcohol mixture used for extracting montan wax from lignite (cf. G.P. 325,165; J., 1920, 814A). The purpose of the alcohol is to take up moisture which might hinder the action of the solvent. Acetone however is also miscible with water, and moreover appears to equal benzol as an extractive agent when applied to Bohemian lignites.—H. J. H.

*Soot etc. in boiler plants.* Donath. See I.

*Oxidation of lignite products.* Schneider. See III.

*Emulsions.* Bechhold and others. See XII.

#### PATENTS.

*Coal washers.* Bureau d'Etudes Ind. F. Courtoy et L. Dubois, and F. Courtoy. E.P. 156,833, 12.6.19.

In a coal washer of the type comprising a trough having one compartment for receiving the coal and another for containing reciprocating pistons for producing suction, the residuary products are removed by suction through valves which are operated directly by the pressure of the washing water and which regulate the suction automatically according to the charge of coal and the proportion of waste required to be removed.—W. E. F. P.

*Coal briquettes; Production of* —. W. Broadbridge, E. Edser, W. W. Stenning, and Minerals Separation, Ltd. E.P. 155,875, 26.8.19.

AN aqueous suspension of coal obtained from colliery waste by the froth flotation process (E.P. 7803 of 1905, F.P. 354,960; J., 1905, 1177) is agitated with liquefied binding medium, e.g., melted pitch, with which the particles become coated so that they may be briquetted. Cresol or kerosene oil may be added to promote frothing and slaked lime to assist flocculation, and the concentration and addition of the binding agent may be carried out in one operation. The crushing of the pitch being unnecessary, soft pitch may be used.—C. I.

*Fuel briquettes from coke waste, coal waste, brown coal, or the like; Manufacture of* —. R. Matsunaga and S. Aoki. E.P. 157,007, 8.3.20.

A MIXTURE of the washed and powdered waste (not more than 2 pts.) with powdered coking bituminous coal (1 pt.) is moistened with water and pressed in moulds, and the briquettes are roasted in an oven. The materials are previously ground to pass a sieve of about 22 meshes to the inch.—W. E. F. P.

*Peat; Treatment of* — D. B. MacDonald, and The Densified Peat, Fuel and Products, Ltd. E.P. 155,895, 20.9.19.

Wet peat is pulped and compressed by forcing it through a reduced aperture. The product is cut into blocks and dried by rapid heating and cooling. It may be used as fuel or as a substitute for wood. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 1216 of 1873, 3536 of 1877, 6704 of 1887, 25,627 of 1897, 2859 of 1901 and 6153 of 1902; J., 1888, 381; 1899, 255; 1903, 485.)—C. I.

*Fuel residues; Process of separating slags containing iron from coke and other* — F. Krupp A.-G. Grusonwerk. E.P. 150,333, 21.8.20. Conv., 26.8.19.

FUEL residues after crushing are passed through highly concentrated magnetic fields, obtained, for example, by means of the separator described in E.P. 100,063 (J., 1916, 694), whereby the slag which almost always contains iron is separated from the unburnt coal and coke. Owing to the complete separation obtained the slag is well suited for the manufacture of building stones.—C. I.

*Coking ovens; Horizontal* — Coke ovens. Soc. Franco-Belge de Fours à Coke. E.P. (a) 125,590, 29.5.19 (Conv., 29.5.18). and (a) 156,034, 12.4.20.

(A) THE following arrangement makes it possible to use both rich and poor gas at the same time in regenerative horizontal coke ovens with vertical heating flues. Two pairs of independent regenerators are provided on either side of the longitudinal centre line of the battery, the inner one of each pair communicating with the odd numbered half-sole flues, the outer one divided into compartments each of which communicates with one of the even-numbered half-sole flues. When heating with coke-oven gas air is admitted to both chimney flues on one side of the battery, is preheated in the regenerators, and the products of combustion pass through the regenerators on the other side. When heating with producer gas the outer regenerator on one side is cut off from the chimney flue and the gas is admitted, while air is admitted to the interior regenerator, and the gas and air pass up alternate flues to unite and burn in the vertical heating flues. As the outer regenerators are divided into compartments, part of the battery may be operated in each way. The cross-sections of the connexions from the chimney flues to the regenerators are proportioned to the distance from the chimney. (a) In coke ovens of the type described under (A) difficulty is met with in forming a hard coke from certain coals owing to the top of the charge not being coked quickly enough. To obviate this the vertical heating flues are arranged in pairs, the air passing up one flue and the products of combustion down the other; this flow is periodically reversed, while the gas is admitted continuously in the upper part between the two flues. The temperature is further controlled by air ports in the pier. Beneath the ovens are two continuous flues, each connected with one regenerator and with alternate heating flues.—C. I.

*Coke oven; Regenerative* — Koksofenbau u. Gasverwertung A.-G. G.P. 327,002, 18.4.16.

In an oven for alternative heating with rich or poor gas, in which the air inlets to two neighbouring heating flues are adjacent to each other and are separated by thick walls from the similarly arranged gas inlets, an equalising flue in the upper part of the regenerator is connected with the air chamber; the air connexions to the heating flues branch off from this equalising flue. These can therefore be so arranged that two air inlets and two gas inlets always lie directly next each other.

The air and producer-gas inlets are always parted by the thick walls so that leakage from one to the other and the production of an explosive mixture is prevented.—C. I.

*Retort-furnace for use in making gas [from vegetable matter]*. B. R. Pfeifer. E.P. 156,029, 20.3.20.

A RECTANGULAR retort set upon a furnace is traversed horizontally by flues carrying the gases of combustion from the fire. The retort is charged with straw or the like through an end door, and the gas passes out through an exit pipe, the tar etc. being removed through the charging door at the end of an operation.—C. I.

*Retort [for making gas from straw]*. G. Harrison and E. G. S. Bennett. U.S.P. 1,363,588, 28.12.20. Appl., 27.3.19.

FOR the production of gas from straw a cylindrical retort is used with a conduit running through it. An intensifier is placed in the conduit, consisting of a pair of spaced perforated walls containing a non-combustible and heat-retaining filling material.—J. H. J.

*Gas manufacture*. G. Helps. E.P. 156,812, 17.5.17.

A gas generator with superheater is combined with a vertical retort placed above it. The air blast in the generator passes through the superheater and heats a steam boiler. The steam blast passes in the reverse direction through the superheater, yields superheated water-gas in the generator and heats the retort. The coke required for the generator is fed in directly from the retort. The heat of the latter may be augmented if necessary by burning part of the water-gas.—C. I.

*Coal or fuel carbonisation and gas-making plant; Combined* — R. and J. Dempster, Ltd., H. S. Knight, and F. J. Siddall. E.P. 157,036, 2.7.20.

A PRODUCER- or water-gas generator is connected with a chamber in which one or more retorts are set, the gas space being filled with checker-work. The generator-gas is at first burnt in this space to effect the distillation of a fuel charge in the retorts, whilst at a later stage the water-gas from the producer after passing round the retorts is led into the latter.—C. I.

*Illuminating gas; Process of obtaining — from lignite*. E.P. Schoch. U.S.P. 1,364,455, 4.1.21. Appl., 30.8.19.

LIGNITE is distilled until the percentage of carbon dioxide in the gas evolved shows a marked decrease, all the gas is collected, and the carbon dioxide removed.—C. I.

*Gas producer and superheater; Combined* — G. F. Rendall, Assr. to Carbon Products Co. U.S.P. 1,366,213, 18.1.21. Appl., 29.6.17.

THE superheater is inside the combustion chamber of the producer and discharges its steam into a chamber below the grate, whence it passes up through the producer. A part of it is used to inject preheated air into the producer from a condenser on the gas main.—C. I.

*[Gas] retort; Continuous vertical — with preheater*. K. Barthel. G.P. 323,837, 26.9.18.

THE heat exchanger or preheater consists of a cylindrical vessel placed above the retort with a hopper interposed and containing a gas-collecting funnel, the mouth of which is of greater cross-section than the retort.—C. I.

*Gas washer and purifier*. G. Coune. E.P. 155,859, 30.4.19.

A GAS washer consists of a fixed horizontal cylin-

der partly filled with liquid and divided into compartments in each of which rotates a washing wheel. Each wheel has a hollow hub conveying the gas, and consists of a double set of rotating elements divided by a disc which also divides the hub. The gas passes first outwards from the axis and then inwards in each compartment.—C. I.

*Gas-epurator [purifier].* V. L. Cazes. U.S.P. 1,364,832, 4.1.21. Appl., 14.11.18.

A HORIZONTAL scrubber has upright baffles attached alternately to the top and bottom. The bottom of the scrubber is perforated and below it is an ash-pit fitted with removable scrapers.—C. I.

*Gas washing and humidifying process.* J. P. Ruth, jun. U.S.P. 1,365,278, 11.1.21. Appl., 16.8.17.

LIQUOR is forced up a cylindrical vessel by centrifugal force or otherwise and the gas is drawn in at a separate inlet in the lower part of the vessel by the partial vacuum produced by the movement of the liquid. The liquid flows over a baffle, passes downward, and is returned to the washing vessel.—C. I.

*Motor fuels.* E. W. Stevens. E.P. 135,514, 21.11.19. Conv., 21.11.17.

A MOTOR fuel contains 50–55 pts. of kerosene or a similar oil, 25 pts. of ethyl alcohol, 5 pts. of fusel oil, 10 pts. of sulphuric or petroleum ether or casinghead gasoline, and 8 pts. of toluol or xylol. In aeroplane fuel the proportion of kerosene is reduced to about 40% of the mixture.—L. A. C.

*Liquid fuel; Burners for —.* E. F. Hooper and J. E. Weyman. E.P. 156,428, 13.2.20.

A SPRAY nozzle for liquid fuel (tar or heavy oil) is controlled by a needle valve operated by a micrometer screw for fine adjustment. The valve may be freely moved on disconnecting the screw. The oil is projected into a mouthpiece of refractory material which has its smallest diameter near the inlet and then diverges outwards. Air is admitted around the edge of a disc which is movable axially away from the inlet of the mouthpiece to vary the opening. The inner surface of the mouthpiece is provided with helical ribs.—W. F. F.

*Oil-shale-treating retort; Vertical —.* J. H. Galloupe, Assr. to The Galloupe Shale Process Co. U.S.P. 1,365,822, 18.1.21. Appl., 18.8.19.

OIL shale is fed into the annular space between two vertical cylinders situated within a cylindrical casing. Flues convey the hot gases from a furnace through the inner cylinder, which is rotated, and also through the space between the middle cylinder and the outer casing.—L. A. C.

*Oils; Process of purifying —.* D. T. Day. U.S.P. 1,365,894, 18.1.21. Appl., 18.2.18.

THE oil is brought into contact with cuprous chloride.—L. A. C.

*Mixing gases [for surface combustion]; Apparatus for —.* Surface Combustion Co., Inc., Asses. of W. B. Eddison. E.P. 117,612, 19.6.18. Conv., 2.7.17.

SEE U.S.P. 1,295,086 of 1919; J., 1919, 566a.

*Gas-generating retort construction.* B. R. Pfeifer, Assr. to The Pfeifer Straw Gas Producer Co. U.S.P. 1,364,801, 4.1.21. Appl., 26.8.19.

SEE E.P. 156,029 of 1920; preceding.

*Coke quenching device.* A. Fränkel. E.P. 157,042, 1.9.20.

See also pages (A) 142, *Bituminous material* (U.S.P. 1,340,855); *Lubricants* (G.P. 326,729). 147,

*Producer-gas and cyanides* (U.S.P. 1,364,838); *Washing gas* (G.P. 328,394). 148, *Hydrogen* (U.S.P. 1,366,185 and G.P. 328,637). 162, *Products from olefines, petroleum, etc.* (U.S.P. 1,365,043–53).

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Charcoal burning; Relation of composition of gases evolved in — to the yield of charcoal.* P. Klason. Brennstoff-Chem., 1920, 1, 79–80.

CHARCOAL burning in heaps is assumed to be equivalent to distillation in a retort at 500° which yields a product of composition equivalent to  $C_2H_4O$ , whereas distillation in a retort at 400° gives a "retort charcoal" which may be represented as  $C_2H_4O$ . Pine wood gives a yield of 37% of charcoal by distillation at 400° C., and 31% at 500° C. The yield is further lowered when charcoal is burnt in the heap by partial combustion. In an actual test the yield was 27%. The gas from charcoal burning in heaps is regarded as a mixture of the gases from the distillation of wood up to 500° C., the  $CO_2$  content of which is 38%, and the products of combustion of  $C_2H_4O$ , which contain 20%  $CO_2$ . On this basis the composition of the gases from pine wood was calculated as  $CO$ , 25.8% (25.6),  $CO_2$  9.3% (9.8),  $(CH_4 + H_2)$  10.7% (9.7),  $N_2$  54.7% (54.9). The figures in brackets were those actually determined. It follows therefore that the  $CO_2$  content of the gases will be high when products of combustion are absent, thus indicating a high yield of charcoal. Actually the percentage of  $CO_2$  in the gases is roughly a measure of the yield of charcoal by weight and therefore a useful method of control of the charcoal-burning process.—H. J. H.

*Softening of carbon.* Gmachl-Pammer. See VII.

*Distillation of exhausted beet slices.* Paar and Starke. See XVII.

### PATENTS.

*Shaft furnace for distillation or carbonisation of waste material, refuse heaps, bituminous shales, and the like [lignite and non-caking coals].* H. Koppers. G.P. 322,547, 29.10.16.

IN a furnace (of the Rolle type) in which the material is made to descend in a layer in contact with the heated iron walls, by means of an inlet perforated for the escape of the gases and vapours produced, the iron heating mantle of the furnace is rotatable in order to prevent local overheating. Ribs on the outer surface of the iron mantle project into grooves in the surrounding masonry, forming a series of heating flues around the iron mantle.—J. H. L.

*Finely divided fuel; Distillation of —.* Bünzlauer Werke Lengersdorff und Co. G.P. 326,604, 2.2.18.

TO effect the distillation of a close-lying fuel, e.g., coal dust, by means of a heated gaseous medium, the generator is arranged in two parts. In the upper part, which can be rotated, a counter current of hot gas acts on the fuel, which reaches the lower part as coke. From the latter gas is generated and serves to effect the distillation of more fuel.—C. I.

*Gas-fire radiants; Manufacture of —.* South Metropolitan Gas Co., and H. Hollings. E.P. 156,416, 21.1.20.

IN the manufacture of gas-fire radiants discoloration of the radiants due to overheating is avoided by substituting for the asbestos usually incorporated a vegetable or animal fibre such as hair, flax, hemp, cotton, or jute. If necessary the vegetable or

animal fibre is subjected to a preliminary washing with dilute hydrochloric acid to remove iron impurities.—J. S. G. T.

*Gas-filled glow discharge lamps; Electric* — A Lederer. E.P. (A) 17,036, 17.7.14, and (a) 17,646, 25.7.14. Conv., (A) 19.7 and (a) 25.7.13.

(A) The electrodes of a gas-filled glow discharge lamp are composed of a solid metal other than the alkali metals, e.g., aluminium, magnesium, tungsten, and a small quantity of one or more of the alkali metals is introduced into the lamp, preferably in the form of a deposit which is not vaporised during operation of the lamp. (B) In a gas-filled lamp as described under (A) the small quantity of alkali metal introduced is confined to the region of the anode or of both electrodes.—J. S. G. T.

### III.—TAR AND TAR PRODUCTS.

*Tar; Estimation of water in* —. Mitteilung des Gasinstituts (Lehr- und Versuchsanstalt). Gas- und Wasserfach, 1921, 64, 71—73.

OWING to the great variations in the percentage of water in tar as it passes through the different receivers in the tar works, samples for estimating the water in tar deliveries should be taken either as the tar flows into the wagons, or from the wagons themselves. In the former case a narrow, valved branch pipe, which should be vertical or sharply inclined, is attached to the delivery pipe some distance from the end, and discharges a continuous sample into a receiver: in the latter a number of samples are withdrawn from the wagons by means of an iron tube, 3—5 cm. diam., fitted with a stopper or valve at the bottom which can be opened and closed by means of an iron rod passing down the tube. Sampling should be done soon after filling the wagon, and the total sample taken by either method must be well agitated before weighing a portion for estimating the water. Three methods are available for the water estimation: fractional distillation, vacuum distillation, and Beck's method (J., 1909, 1027). Distillation methods have the advantage that water, naphthalene wash-oil, and benzol wash-oil are estimated in one operation. The tar is distilled in a copper flask fitted with a Liebig condenser until the oily distillate is free from water, i.e., about 200° C., and the volume of water is read off direct in a measuring cylinder. The addition of 10% of xylol and some pumice stone to the tar facilitates the distillation and prevents bumping. Decomposition of the tar is reduced if the distillation is carried out under vacuum, but frothing of the contents of the flask renders this method somewhat difficult to operate. The tar is first heated until it begins to boil under ordinary pressure, and the distillation is then carried out without further heating by gradually raising the vacuum to about 700 mm. Beck's method is more rapid in operation than the other methods and gives results in very close agreement with those obtained by fractional distillation.—L. A. C.

*Tar; Technical chemical analysis of* —. P. Falcicola. Giorn. Chim. Ind. Applic., 1920, 1, 38—45. (Cf. J., 1917, 638.)

For sampling tar a dipping cylinder similar to that employed for crude glycerin (J., 1911, 556) may be used. In determining the moisture content of tar by distillation, bumping and excessive frothing may be avoided by heating 80—100 g. of the tar in a 700—800 c.c. glass flask fitted at the neck with two large glass bulbs containing a thermometer. An annular burner is used and is placed first on a level with the surface of the tar, using small flames; it is then gradually lowered and the flame increased until these reach the bottom of the flask. Sulphur in

tar may be estimated as follows: About 0.5 g. of the tar is intimately mixed by means of two spatulae with about 15 g. of a mixture of 4 pts. of potassium chlorate, 1 pt. of potassium nitrate, and 1 pt. of ammonium nitrate. The mixture is introduced into a test-tube and this supported vertically in a very tall beaker by means of glass beads or the like. This beaker is placed in a still wider and taller one, a fuse in contact with the mixture being ignited. The test-tube and its containing cylinder are then covered with a tall heavy glass cylinder with its edge notched with file marks, while about 500—600 c.c. of water containing sodium hydroxide and a few drops of bromine water is placed in the beaker. The combustion of the mixture proceeds gradually and lasts several minutes, the gases emitted being washed by the alkaline liquid sealing the cylinder. When the apparatus has cooled the cylinder is removed and washed, the washings, as well as those of the test-tube and beads, being added to the liquid decanted from the beaker. The whole liquid is then acidified with hydrochloric acid, concentrated, filtered, and precipitated hot with barium chloride. The small amount of carbonaceous residue sometimes left on the filter does not invalidate the results. For tars, bitumens, and petroleum this method gives results slightly lower than those obtained by the Carius method.

—T. H. P.

*Lignite products; Action of oxygen under pressure on* —. W. Schneider. Brennstoff-Chem., 1920, 1, 70—72, 80—85.

MOLECULAR oxygen, especially in the form of air, has certain advantages, both financial and technical, over oxidising agents in common use. The velocity of reaction can be varied within wide limits by changes of pressure and temperature. Tubular autoclaves have been constructed and used for effecting oxidation under a pressure of 70 atm. and at 200° C. The contents could be sampled so as to permit the study of the course of the reaction. In such apparatus it was attempted to prepare fatty acids and soaps from the products obtained from lignite. The humic portions, though quantitatively most important, are unsuitable as starting material owing to their cyclic structure. The bitumen compounds, containing long aliphatic chains and therefore presumably a more promising raw material, were not so satisfactory in practice as the tar oils. Viscous acid substances, amounting to 55% of the oil taken, were obtained by oxidising lignite tar oil in presence of alkalis and acidifying the product. These acids were presumably polynaphthenic acids. Commercial paraffins containing compounds with about 25 carbon atoms were oxidised and yielded mainly monobasic acids with about 13—19 carbon atoms and small quantities of dibasic acids and other compounds, e.g., alcohols and aldehydes, presumably intermediate compounds. The influence of conditions on oxidation—e.g., temperature, pressure, concentration, catalysts, and time—was studied in the experiments on paraffins, and the behaviour of paraffin when heated in presence of alkali at 170° C. and 30 atm. pressure was taken as a standard of comparison. The salts of the fatty acids obtained seemed to serve as satisfactory soaps. Montan wax, though oxidised by ozone at air temperature, proved more refractory towards compressed oxygen than paraffins. Fatty acids were formed, but presumably so slowly that they were extensively broken down to small molecules, so that no large yields of soap-forming products were obtained.—H. J. H.

*Benzene and benzaldehyde; Mode of pyrogenic decomposition, at high temperature, of* —. E. Peytral. Bull. Soc. Chim., 1921, 29, 44—47.

In the pyrogenic decomposition of benzene at high temperatures the principal change is the formation

of diphenyl and hydrogen. At the same time some methane is formed by more profound decomposition either of the benzene or of the diphenyl. In the decomposition of benzaldehyde there are two primary changes, namely,  $C_6H_5.CHO = C_6H_5 + CO$  and  $2C_6H_5.CHO = C_6H_5.C_6H_5 + 2CO + H_2$ . At the same time a small amount of the benzaldehyde is reduced by the hydrogen giving anthracene, and some of the diphenyl is decomposed giving methane.

—W. G.

**$\Delta^1$ -Dihydronaphthalene.** III. Modes of formation of  $\Delta^1$ -dihydronaphthalene. F. Straus and L. Lemmel. Ber., 1921, 54, 25–49.

THE amounts of  $\Delta^1$ - and  $\Delta^2$ -dihydronaphthalenes in mixtures of these substances with naphthalene and tetrahydronaphthalene can be estimated by means of mercuric acetate which oxidises the first-named with production of mercurous acetate in accordance with the scheme:  $C_{10}H_8 + 2(CH_3CO)_2Hg + 2H_2O = C_{10}H_8(OH)_2 + 2CH_3CO_2Hg + 2CH_3CO_2H$ , forms an additive compound with the  $\Delta^1$ -derivative which is soluble in benzene, and does not react with naphthalene or its tetrahydro compound. Isomerisation of the  $\Delta^1$ - to the  $\Delta^2$ -isomeride under the influence of alcoholic sodium ethoxide solution occurs with greater readiness than was previously supposed. In the preparation of  $\Delta^1$ -dihydronaphthalene by the action of sodium and ethyl alcohol on naphthalene there is a partial isomerisation of the  $\Delta^1$ - into the  $\Delta^2$ -derivative, which is thus always present to the extent of about 5%, and if, after solution of the sodium is complete, the final concentration of the alkoxide is raised by distilling off a portion of the alcohol, the isomerisation may be made to occur quantitatively, thus giving the simplest method of preparing  $\Delta^1$ -dihydronaphthalene, which can be separated from admixed naphthalene and tetrahydronaphthalene by conversion into the dibromide from which the hydrocarbon is readily regenerated. The dihydronaphthalene obtained from *ac*-tetrahydro- $\beta$ -naphthol,  $\beta$ -chlorotetrahydronaphthalene, and *ac*-tetrahydro- $\beta$ -naphthylamine nitrite, is the  $\Delta^1$ -compound and not the  $\Delta^2$ -derivative as supposed previously. The dihydronaphthalenes are conveniently reduced to tetrahydronaphthalene by hydrogen in alcoholic solution in the presence of palladium; under these conditions, the second nucleus remains unattacked. (Cf. J.C.S., Mar.)—H. W.

**Emulsions.** Bechhold and others. See XII.

#### PATENTS.

**Anthracene; Process for the purification of crude** —. Kinzberger and Co. E.P. 144,656, 8.6.20. Conv., 31.3.17. Addn. to 144,648.

In the process described in the chief patent (J., 1921, 5A), the use of a large excess of potassium hydroxide avoids the necessity of removing excess of water by distillation and enables the quantitative formation of potassium-carbazole to take place at a lower temperature, e.g., 120° C.—L. A. C.

**Fusions with molten caustic soda or caustic potash; Method of conducting** —. B. D. Steele and R. B. Robertson. E.P. 156,269, 9.2.18.

A hot concentrated solution of a sulphonate, e.g., sodium benzenesulphonate, is fed slowly on to the surface of molten sodium or potassium hydroxide.

—L. A. C.

**Bituminous material; Composition of matter containing** —. J. M. Weiss, Assr. to The Barrett Co. U.S.P. 1,340,855, 18.5.20. Appl., 8.11.19.

A composition suitable as a fuel for Diesel engines, for treating the surfaces of roads, and as a paint or the like, is composed of about 80 pts. of pitch, 20 pts. of petroleum naphtha, and 1–3 pts. of tar acids. The addition of the tar acids causes the

mixture to form a colloidal solution or suspension showing practically no tendency to separate.

**Fumes; Removing** — from [bituminous] materials. K. B. Howell, Assr. to The Barrett Co. U.S.P. 1,348,140, 27.7.20. Appl., 23.12.18.

A cooling medium is passed over hot bituminous material, e.g., pitch, which is being comminuted to convert it into filamentary form. Substances that are volatile at the temperature of operation are carried away by the cooling medium which circulates in a closed circuit including a condenser.

**Cresols; Separation of meta- and para-** —. C. R. Downs and R. S. Potter, Assrs. to The Barrett Co. U.S.P. 1,364,547, 4.1.21. Appl., 10.4.19.

A mixture containing *m*-cresolsulphonic acid and *p*-cresol is distilled *in vacuo* in the presence of steam at such a temperature that *p*-cresol distills, but the *m*-cresolsulphonic acid is not appreciably decomposed.—L. A. C.

**Benzanthrone; Method of purifying** —. L. C. Daniels, Assr. to National Aniline and Chemical Co. U.S.P. 1,365,024, 11.1.21. Appl., 9.1.20.

BENZANTHRONE is dissolved in hot halogen derivatives of aromatic hydrocarbons, insoluble impurities are separated, and the solution is cooled, whereby benzanthrone is deposited from solution.

—L. A. C.

**Phenols from red-wood [tar]; Production of** —. W. J. Hund. U.S.P. 1,365,407, 11.1.21. Appl., 25.4.17.

THE tar obtained by distilling resinous red-wood above 450° C. is distilled, and the oily fraction collecting between 150° and 250° C. is extracted with an alkali hydroxide solution to dissolve phenolic compounds which are afterwards separated from the solution.—L. A. C.

**Aromatic hydrocarbons; Process of producing** —.

A. S. Ramage, Assr. to Bostaph Engineering Corp. U.S.P. 1,365,849, 18.1.21. Appl., 24.6.18.

AN alkyl group in an aromatic compound containing one or more alkyl groups is replaced by hydrogen by treating the aromatic compound with hydrogen at 700° C. in the presence of a lower oxide of iron as catalyst.—L. A. C.

**Lubricants, resins, etc.; Manufacture of hydrocarbons serving as** —. Badische Anilin- u. Soda-Fabr. G.P. 326,729, 9.6.18.

By the interaction of xylol and ethylene chloride in the presence of aluminium chloride, products free from chlorine are obtained either as a viscous liquid mixture of high flash point or a solid resin.

—C. I.

**Washing oils etc.; Continuous process and apparatus for distillation of light products from** —. C. H. Borrmann. G.P. 326,730, 25.11.17.

THE preheated oil is directly heated in a system of pipes under atmospheric pressure, to a temperature above the highest boiling point of the light products desired, so that a quantity of heavy oil also comes over. The vapour produced serves for driving off the main part of the light oil in a continuous still, the crude light oil being freed from the higher boiling fractions in a directly connected fractionating column. By a variation the process can be used for the separation of the tarry residue from washing oil.—C. I.

**Heater for obtaining hot air with reduced oxygen content.** U.S.P. 1,352,481. See I.

**Resins.** U.S.P. 1,365,423. See XIII.





*Fabrics, especially paper-yarn fabrics; Impregnation of —.* O. Ruff. G.P. 327,399, 27.10.17.

Fabric impregnated with a phenol, such as cresol, formaldehyde, and an alkali, is heated in an atmosphere containing carbon dioxide. The change of the caustic alkali to carbonate is favourable to the formation of the water-insoluble phenol-formaldehyde condensation product, and both the dry and wet strengths of the fabric are increased.—A. J. H.

*Cellulose acetate; Manufacture of solutions of —.* Badische Anilin- und Soda-Fabrik. E.P. 145,511, 22.6.20. Conv., 9.2.14.

CELLULOSE acetate soluble in acetone is dissolved in a fully hydrogenised monocyclic ketone with or without the addition of suitable diluents. For example, 4 pts. of cellulose acetate is dissolved in 96 pts. of cyclohexanone to give a solution suitable for lacquering and drawing to artificial silk.

—A. J. H.

*Plastic [nitrocellulose composition].* L. E. Barton and H. A. Gardner, Assrs. to Titanium Pigment Co. U.S.P. 1,365,882, 18.1.21. Appl., 7.2.17.

THE composition contains nitrocellulose, titanium oxide, and camphor.—A. J. H.

*Larch; Recovery of organic products from —.* H. F. Weiss, Assr. to C. F. Burgess Laboratories. U.S.P. 1,364,418, 4.1.21. Appl., 14.5.18. (Cf. U.S.P. 1,358,129; J., 1921, 6A.)

WOOD of the western larch is comminuted and digested with sulphite liquor to recover sulphite pulp and a waste liquor containing soluble constituents of the larch, including conversion products of galactan.—J. H. L.

*Cellulose; Process for separating — from wood and other materials containing cellulose.* R. Hartmuth. G.P. 326,705, 9.7.19.

THE raw material is heated with phenols, or substances having a phenolic character, in a concentrated form or diluted with water, alcohol, benzene, benzene derivatives, or aliphatic hydrocarbons, in the presence of a catalyst. A suitable catalyst is 0.01% of hydrochloric acid. The lignin combines with the phenol to form a soluble substance which may be used as an adhesive, lacquer, or the like, while the cellulose remains white and unchanged.

—A. J. H.

*Cellulose; Process for obtaining — from wood and similar materials containing cellulose.* W. Schlosser. G.P. 328,729, 27.9.19.

THE raw material is treated either hot or cold with primary and secondary aliphatic and aromatic amines, either alone or diluted with alcohol or benzene etc. The reaction proceeds more rapidly in the presence of a condensing agent such as hydrochloric acid or zinc chloride.—A. J. H.

*Cellulosic materials; Process for the disintegration of —.* R. Schwarzkopf. G.P. 328,730, 27.9.19.

THE raw material is treated either hot or cold with acid chlorides, such as sulphuryl chloride, thionyl chloride, phosphorus oxychloride, acetyl chloride, benzoyl chloride, etc., which may be diluted with indifferent solvents, such as benzene and carbon tetrachloride. In cases where the acid chlorides do not dissolve the non-cellulosic substances directly the cellulose may be purified by an after-treatment with alkalis.—A. J. H.

*Parchment paper; Manufacture of —.* C. G. Schwalbe. G.P. 303,305, 23.6.17.

BEFORE being made into paper the raw material, such as cellulose, half-stuff, mechanical wood pulp, etc., is treated in the edge runner or in the hollander with oxidising agents, such as oxides of

nitrogen, permanganates, peroxides, perchlorates, or perborates. By this means parchmentisation is effected and a product of increased tenacity is obtained.—A. J. H.

*Cigarette-paper; Manufacture of —.* Zellstoff-fabr. Waldhof. G.P. 327,735, 29.11.17.

SULPHITE pulp to be used for the manufacture of cigarette-paper is treated with alkalis whereby its unpleasant taste is removed.—A. J. H.

*Fermentable matter; Apparatus for recovering volatile and — from liquids [sulphite-cellulose waste lyes].* A. Schaefer, Assr. to Northwest Process Co. U.S.P. 1,342,721, 8.6.20. Appl., 25.1.17.

AN evaporating chamber contains a stacked coil of steam pipes surmounted by a perforated distributor which distributes the liquor over the pipes in the form of a thin sheet. A valved discharge pipe at the bottom of the evaporating chamber communicates with a residuum chamber below, in which the liquor is neutralised and from which it is led to the fermenting vats. A saturation tank, in which the sulphur dioxide evolved is absorbed in water, is connected with the evaporating chamber and also with a vacuum pump which maintains a reduced pressure in the evaporator.—J. H. L.

*Preparing cellulose acetate for dyeing.* U.S.P. 1,366,023. See VI.

*Celluloid.* U.S.P. 1,364,342. See XXI.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Sulphur dyes; Process for rapidly developing the full bright tone of fabrics dyed with —.* E. Justin-Mueller. Soc. Ind. Mulhouse. Sealed Note No. 1804, 4.11.08. Rev. Gén. Mat. Col., 1921, 25, 5.

THE dyed fabric is treated for 10–20 mins. in a cold bath containing 2–3% of sodium disulphite of 38° B. (sp. gr. 1.36) and 1–1½% (on the weight of fabric) of caustic soda of 38° B., mangled and dried without washing. The bath must be alkaline to Brilliant Yellow test paper.—A. J. H.

*Sulphur dyestuffs; Process for printing coloured discharges by means of —.* E. Justin-Mueller. Rev. Gén. Mat. Col., 1920, 24, 85–86.

A SUITABLE printing paste is prepared by mixing 20 c.c. of caustic soda of 38° B. (sp. gr. 1.36) with the dyestuff, adding 250 c.c. of boiling water and 100 g. of dextrin, and heating the mixture for 20–30 mins. on a water bath until the reduction of the dyestuff is complete. For difficultly reducible dyes, the addition of 20 g. of sugar, glucose, or molasses etc. is necessary. For printing on white grounds, the mixture is diluted to 1 l. with neutral starch thickening containing 15 g. of Hydraldite C extra. A greater amount of Hydraldite must be used for the discharging of grounds dyed with azo dyestuffs. Good results are only obtained if the printing paste is not strongly alkaline and if the reducing agent used is not easily oxidisable. The printed fabric is steamed for 3–4 mins. at 102° C., washed, and soaped.—A. J. H.

*Battick printing.* L. Kollmann. Textilber., 1920, 1, 149–151. Chem. Zentr., 1921, 92, II., 35–36.

THE influence of the previous preparation of cloth for battick printing has been investigated. Mer-cerisation affects not the nature but only the depth of shade of the ground and grain. Alkaline treatment gives a clear vigorous grain. For "throwing up" wax reserves, gums and dextrins are satisfactory. Paraffin-colophony wax reserves are



best printed at 75°–80° C. in order to obtain clear effects. In breaking, the reserve must be actually broken and not merely bent. Breaking is best conducted under cold water since reserves broken in air tend to re-unite and so render dyeing more difficult. Dyeing should quickly follow breaking. The mercerisation of waxed fabrics allows special effects to be obtained.—A. J. H.

## PATENTS.

*Bleaching treatment of open fabrics; Apparatus for —.* E. W. Hunt. E.P. 156,359, 25.10.19.

THE fabric is plaited down full width into a wagon having a perforated bottom, and pressed down by means of an open grid cover. The two ends of the wagon are then screwed towards each other so that the fabric is compressed. The wagon is run into a horizontal type of kier and liquor is passed upwards through the fabric.—A. J. H.

*Dyeing textile fabric; Method and product for —.* J. W. Fries. U.S.P. 1,341,637, 1.6.20. Appl., 13.6.19.

A MIXTURE of indigo paste and logwood extract can be prepared in the form of a dry powder soluble in soap solution and in alizarin (Turkey-red) oil. The logwood extract should be freshly made, or, if in an oxidised condition, should first be treated with a small quantity of yeast. Fabrics are dyed by padding or immersion in a solution of the mixed dye-stuff and the colour is developed by treatment with salts of copper, iron, or nickel.

*Fabric [silk and the like] pieces; Plant for treatment of —.* F. J. B. Knibiebler. U.S.P. 1,365,175, 11.1.21. Appl., 5.12.19.

THE machine consists of a number of compartments which may be separated or not as desired and which contain the fabric to be treated. Conduits are arranged so that a controlled amount of liquid is delivered to each compartment, and means are provided for guiding the fabric therein.—A. J. H.

*Dyeing machine.* E. W. Morgan. U.S.P. 1,365,936 18.1.21. Appl., 11.10.20.

A BARREL is provided with perforated partitions on opposite sides of its axis and liquid is sprayed on to the goods in the middle part of the barrel. Means are provided for revolving the barrel intermittently and for holding it stationary while the liquid is being sprayed on to the goods. A steam heating device is arranged round the barrel.—A. J. H.

*Cellulose; Process for preparing — for dyeing.* E. Prince, Assr. to Soc. Chim. des Usines du Rhône (anc. Gilliard, P. Monnet et Cartier). U.S.P. 1,366,023, 18.1.21. Appl., 25.6.20.

CELLULOSE acetate is treated before dyeing with a relatively concentrated aqueous solution of alkali salts to which an alkali has been added.

—A. J. H.

*[Cotton-silk and cotton-wool] union dyeing with sulphur dyestuffs; Process for — whereby the wool or silk remains unstained.* Farbenfabr. vorm. Bayer und Co. G.P. 326,649, 18.9.14.

BEFORE dyeing, the union fabric is treated with tannic acid and a fixing agent such as tartar emetic or formaldehyde.—A. J. H.

*Fast shades on vegetable fibres; Production of — and [azo] dyestuffs therefor.* H. Levinstein and J. Baddiley. U.S.P. 1,365,706, 18.1.21. Appl., 14.2.16.

SEE E.P. 8569 of 1914; J., 1915, 709.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*[Sulphuric acid manufacture;] The chamber process [for —] under tropical conditions.* W. H. Mawdsley. Austral. Inst. Min. and Met. Chem. Trade J., 1921, 68, 133–134.

A PLANT for the chamber process at Mount Morgan consisted of 7 chambers, 1 Glover, 2 Gay-Lussac towers, and 1 pipe column, the whole being run as one unit, and the furnace comprised one brimstone burner and four pyrites kilns. Records of running during one winter month, and one summer month, showed yields of 91.40 and 86.6% respectively, calculated on sulphur burned. To examine the effect of the acid on lead, strips of English and Australian lead, of best "chemical" quality, were suspended in each chamber; the average losses per annum were 11.9 and 11.2%, and total losses in three years were 29.49 and 27.72% respectively. "Creeping" of the lead was a source of trouble; it was to some extent obviated by rolling the sheets thicker for 2 ft. from the end. To support the lead when creeping had begun, provision was made to allow the horizontal supporting rails to be lowered. A device used for obtaining a fair sample of acid from different layers consisted of a pipe fitting closely in a copper cylinder, both pipe and cylinder having a vertical row of holes. These holes were caused to register for the purpose of taking a sample; and the pipe was turned so as to close the holes in the cylinder as the sample was withdrawn.

—W. J. W.

*Arc process [for fixation of nitrogen]; Power cost in the —.* T. C. Hagemann. Chem. Trade J., 1921, 68, 170.

ALLOWANCE being made for auxiliary use of power and losses in absorption, the yield per k.w.-year in the arc process of nitrogen fixation amounts to only 115 kg. of fixed nitrogen compared with 450–500 kg. yielded by the cyanamide process. Graphs are given showing the cost of energy in the arc process as a percentage of the market price, the latter being taken as either £150, £100, or £73 16s. per ton of combined nitrogen. Cheap water power is taken as costing £1 4s. per k.w.-year, average cost of water power is estimated at £2 per k.w.-year, and steam-generated power at £3 15s. per k.w.-year. With an average pre-war price of £2 per k.w.-year for power, power charges amounted to 23.5% of the selling price, £73 16s. per ton of combined nitrogen. At a power cost of £2 6s. per k.w.-year, power charges amount to 15% and 20% respectively of the market prices, £150 and £100 per ton of combined nitrogen. In the arc process for manufacturing calcium nitrate, 15% of the yield is obtained as sodium nitrite, the market price for the combined nitrogen of which is about 67% more than of that in calcium nitrate.

—J. S. G. T.

*Hydrocyanic acid; Preparation of anhydrous —.* K. Ziegler. Ber., 1921, 54, 110–112.

GATTERMANN'S process is modified by the replacement of potassium ferrocyanide by sodium cyanide; with the apparatus described, about 1 kg. of sodium cyanide can be used at a time, the yield of hydrocyanic acid being 90% of that theoretically possible and the cost about  $\frac{1}{3}$ – $\frac{1}{4}$  of that of the older method. A glass flask of 5 l. capacity is provided with a rubber stopper with three holes. The first of these serves for connexion with the Gattermann air condenser and apparatus; the second holds a dropping funnel and the third a mercury safety-tube to which a second dropping funnel is attached by means of a side-tube. The ends of the dropping funnels are brought close together and immediately

above a small filter funnel which is fastened to the underside of the stopper by a stout wire; the stem of the funnel is bent in the form of a U and cut off so that the end is 0.5–1 cm. below the rim of the funnel. Concentrated sodium cyanide solution and an equimolecular amount of sulphuric acid (1:1) are allowed to flow into the funnel simultaneously; hydrocyanic acid is evolved immediately and uniformly and reaction is practically complete in the funnel itself, so that fresh solutions are continuously brought into contact with one another, whilst the bisulphate solution drops into the flask. After addition of the reagents is complete, the remainder of the hydrocyanic acid is driven into the receiver by heating the contents of the flask to the boiling point. Anhydrous hydrocyanic acid can be preserved unchanged for months if sealed in a glass vessel with a little calcium chloride.—H. W.

*Halogen compounds; Dissociation of* —. A. von Weinberg. *Z. Physik*, 1920, 3, 337–342.

The heats of dissociation of the following alkali salts have been calculated as: LiCl, 193.2; LiBr, 150.1; LiI, 129.0; NaCl, 181.4; NaBr, 140.1; NaI, 120.9; KCl, 183.9; KBr, 144.2; KI, 126.6; RbCl, 183.8; RbBr, 145.0; RbI, 127.9; CsCl, 184.2; CsBr, 145.8; CsI, 129.8; and the heats of sublimation as NaCl, 48.8; NaBr, 45.7; NaI, 43.3; KCl, 48.2; KBr, 45.2; KI, 44.9. (*Cf. J.C.S., Mar.*)—J. F. S.

*Aluminium nitrate; Hydrates of* —. K. Inamura. *Mem. Coll. Sci., Kyoto*, 1920, 4, 105–112.

EXPERIMENTS on the equilibrium in the system  $\text{H}_2\text{O}-\text{Al}(\text{NO}_3)_3-\text{HNO}_3$  at 25° C. showed that three hydrates of aluminium nitrate exist, with 18 $\text{H}_2\text{O}$ , 16 $\text{H}_2\text{O}$ , and 12  $\text{H}_2\text{O}$  respectively. The first is stable in nitric acid below 73% in strength, the second in 73–81% acid, and the last in acid stronger than 81% up to 90.5%, the limit of the experiments. A curve is given showing the rapid fall in solubility of aluminium nitrate in nitric acid of increasing strength up to 60%, from which point the subsequent change is slight.—E. H. R.

*Basic exchange in silicates.* E. Ramann and H. Junk. *Z. anorg. Chem.*, 1920, 114, 90–104.

In continuation of previous work (J., 1916, 1129; 1919, 174 A) the formation of magnesium permutite by the action of magnesium salts on ammonium, sodium, and potassium permutites was studied. As in other cases, the reaction is ionic, and there is no evidence of physical adsorption. Pure magnesium permutite could not be prepared, not more than half of the bases present in the permutite being replaced by magnesium. The whole of the ammonium could not be displaced from ammonium permutite by treatment with carnallite ( $\text{MgCl}_2+\text{KCl}$ ) or kainite ( $\text{MgSO}_4+\text{KCl}$ ) mixtures. Mixed salt solutions decompose the permutite to some extent, especially solutions containing magnesium or ammonium.—E. H. R.

*Iron oxides; Reversible reactions of carbon monoxide with* —. G. Chaudron. *Comptes rend.*, 1921, 172, 152–155.

FERRIC oxide is reduced to the magnetic oxide by carbon monoxide. Beyond this stage the equilibrium depends on the temperature. Below 580° C. there is a single system corresponding to the equation  $\text{Fe}_2\text{O}_3+4\text{CO}\rightleftharpoons 3\text{Fe}+4\text{CO}_2$ . Above 580° C. there are two equilibria before arriving at iron, namely,  $\text{Fe}_2\text{O}_3+\text{CO}\rightleftharpoons 3\text{FeO}+\text{CO}_2$ , and  $\text{FeO}+\text{CO}\rightleftharpoons \text{Fe}+\text{CO}_2$ . The three solid phases co-exist in equilibrium at 580° C. Below 580° C. ferrous oxide is unstable, and is transformed into a mixture of iron and the magnetic oxide,  $4\text{FeO}\rightleftharpoons 3\text{Fe}_2\text{O}_3+\text{Fe}$ .—W. G.

*Thorium hydride; Existence of a gaseous* —. A. Klauber and J. M. von Mollenheim. *Z. anorg. Chem.*, 1920, 113, 306–316.

By a method similar to that employed by Paneth and others for detecting hydrides of lead and bismuth (J., 1920, 747 A), the existence of a gaseous hydride of thorium has been established. In a Marsh tube the hydride is decomposed, forming at first a dark brown deposit, afterwards changing to dark grey with a metallic appearance. The deposit was identified as thorium by its reactions. The hydride is very unstable, and its composition could not be determined.—E. H. R.

*Protactinium; Properties of* —. II. *Life period and content in uranium minerals.* O. Hahn and L. Meitner. *Ber.*, 1921, 54, 69–77.

THE half-life period of protactinium has been calculated by separating the latter from uranium salts of approximately known age (three preparations, one of the nitrate at least 20 years old, the second of the nitrate at least 52 years old, and the third consisting of a mixture of yellow uranium oxide and uranium acetate each about 60 years old) and measurement of its activity in comparison with that of uranium, the mean of three concordant experiments being about 12,000 years. This value is to be regarded as the lower limit. From this datum and the known disintegration ratio of the actinium to the uranium series it is calculated that a given uranium mineral contains 72 mg. of protactinium per ton of uranium, the corresponding figure for radium being 330 mg. (*Cf. J.C.S., Mar.*)—H. W.

*Carbon; Softening of* —. J. Gmachl-Pammer. *Monatsh.*, 1920, 41, 467–476.

THE results of the author's experiments on the behaviour of carbons of various kinds when heated electrically show that carbon containing impurities may be made to bend at a distinctly, although not greatly, lower temperature than the pure element. Graphite rods soften only at a far higher temperature than arc carbons, confirmation of this observation being found in the fact that protracted heating of arc carbons, which converts the latter into graphite, raises the softening point to a marked extent.—T. H. P.

*Decomposition of formic acid.* Muller and Peytral. *See XX.*

*Precipitation of iron from hydrochloric acid solution.* Patten and Mains. *See XXIII.*

*Chromic acid.* Kolthoff. *See XXIII.*

#### PATENTS.

*Sulphuric acid; Manufacture of* —. South Metropolitan Gas Co., and P. Parrish. *E.P.* 156,328, 6.10.19.

To promote reaction between the sulphurous gases and the nitrous vitriol, the latter is passed through a closed tank, which is subdivided into compartments by partitions depending from the roof. The lower edges of these partitions are serrated and are immersed in the nitrous vitriol to a depth not exceeding 4 ins., the level of the acid being kept just above the recesses of the serrations. The sulphurous gases enter at one end of the tank and, after bubbling under each partition, escape at the other end.—W. J. W.

*Sulphuric acid; Process for raising the efficiency of* —. *Reed.* U.S.P. 1,363,918, 28.12.20. Appl., 21.8.18.

CONCENTRATED sulphuric acid is produced by heating a mixture of air, sulphur dioxide, and a nitrogen oxide, absorbing the gaseous products of reaction in concentrated sulphuric acid, and then removing the nitrogen oxides from it.—W. J. W.

*Sulphuric acid; Process for raising the efficiency of plant for manufacture of — by the contact process.* M. Leeck. G.P. 307,092, 16.1.18.

ONE fan is placed between the burners and the purification plant for the sulphurous gases, and a second between the contact chamber and the absorption plant. Through the double control the pressure and the gas composition can be regulated as desired in all parts of the plant.—C. I.

*Sulphuric acid; Apparatus for concentration of —.* S. Barth. G.P. 304,343, 14.7.15.

THE concentration vessel has, throughout its whole length, cross divisions over which the acid flows, the division walls being provided with perforations opposite each other. The sections formed in this way are connected by separate pipes with the gas exit main, and are also provided with side openings for cleaning.—C. I.

*Ammonia; Direct oxidation of —.* L. Duparc and C. Urfer. E.P. 133,041, 24.9.19. Conv., 24.9.18.

A MIXTURE of 4 vols. of ammonia and 7 vols. of oxygen is passed in an undried condition, at a comparatively high velocity, over a catalyst of rhodium black or asbestos containing 3–5% of rhodium, the reacting gases being heated to 500°–650° C. The formation of ammonium nitrate and nitrite is thus avoided, and a yield of 97%–100% of nitrogen peroxide is obtained.—W. J. W.

*Lead sulphate; Process for making —.* P. A. Mackay. E.P. 155,945, 18.10.19.

AT an initial temperature of 150° C., lead is rapidly dissolved by oleum, and when reaction has begun, complete conversion into lead sulphate takes place without further application of heat, or at least at a temperature not exceeding 100° C. If the lead is first coated with an electro-negative metal, such as mercury or copper, by treatment with a metallic salt, or if such metals or their salts are added to the oleum during the operation, the working temperature is reduced to 90°–100° C., with violent exothermic reaction. The lead sulphate is precipitated by diluting the solution.—W. J. W.

*Sulphur dioxide; Composition for liberating —.* L. G. Wesson. U.S.P. (A) 1,356,029 and (B) 1,356,030, 19.10.20. Appl., 31.5 and 12.6.19.

(A) A MIXTURE which is stable while dry but decomposed by damp air, is composed of a solid, hygroscopic acid material and a solid metabisulphite. (B) A mixture of a dry solid pyrosulphate and bisulphite or metabisulphite.

*Carbides; Apparatus for the production of nitrogen compounds from metal —.* V. Thrane. U.S.P. 1,364,157, 4.1.21. Appl., 9.1.18.

A FURNACE for treating carbides with nitrogen comprises a compartment within which is a movable table, and means are provided for delivering a layer of inert material upon the table and a layer of carbide upon the inert material, and for passing a current of nitrogen into the chamber in a direction opposite to that in which the table moves.—J. H. L.

*Double sulphates [sodium ammonium sulphate]; Process of splitting — and washing the products.* G. N. Vis. U.S.P. 1,364,822, 4.1.21. Appl., 18.12.18.

SODIUM ammonium sulphate is dehydrated, heated in a solution of ammonium sulphate, the precipitated anhydrous sodium sulphate is removed, and the solution crystallised, giving ammonium sulphate mixed with about 10% of sodium sulphate.—C. I.

*Ammonium sulphate; Process for transforming free or combined ammonia into —.* G. N. Vis. U.S.P. 1,366,301, 18.1.21. Appl., 25.10.18.

AMMONIA (free or combined) mixed with inert gas or steam is absorbed by means of sodium bisulphate practically free from uncombined sulphuric acid to obtain double sodium ammonium sulphate, and the resulting solution is diluted and cooled, whereby sodium sulphate crystallises out as  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ .

*Gas; Washing of — with acid for ammonia recovery etc.* C. Still. G.P. 328,394, 9.8.19.

IN an ammonium sulphate saturator with several vertical pipes dipping into a single vessel, each pipe has a constriction a short distance above the surface of the liquid, at which point any pipe can be shut off as desired.—C. I.

*[Producer] gas and cyanides; Manufacture of —.* R. Franchot, Assr. to Ferro Chemicals, Inc. U.S.P. 1,304,838, 4.1.21. Appl., 8.7.19.

PRODUCER gas is generated at a temperature sufficient to slag the ash in the fuel and, without cooling, is passed over a charge of shaped pervious masses formed of iron, carbon, and a relatively small proportion of alkali carbonate.—C. I.

*Ammonia [from cyanamides]; Preparation of —.* W. O. Snelling. U.S.P. 1,365,739, 18.1.21. Appl., 24.3.16.

A SUSPENSION of the cyanamide in cold water is brought into heat-interchanging relation with the hot reaction products of a preceding operation on a similar suspension.—W. E. F. F.

*Chlorates, bromates, and iodates; Manufacture of —.* H. Schulz. G.P. 328,211, 15.6.15.

HALOGENS and air at atmospheric or increased pressure are passed into an aqueous suspension of any suitable oxide or hydroxide in presence of a catalyst, such as manganese dioxide; or air may be passed into a solution in which the catalyst is suspended and in which a halogen is being generated. A high yield of chlorates etc. is obtained, whereas without the use of air or a catalyst, only one-sixth of the halogen is converted into chlorate, bromate, or iodate.—C. I.

*Hydrogen peroxide; Method of making —.* R. Jacquelet. U.S.P. 1,364,558, 4.1.21. Appl., 19.11.19.

BARIUM peroxide is added to a weak solution of hydrochloric acid, and nitric acid equivalent to the hydrochloric acid is then added to the mixture. This process is repeated until hydrogen peroxide solution of the desired strength, containing a small amount of barium chloride, is obtained.—W. J. W.

*Boron carbide; Manufacture of — especially in shaped pieces.* E. Podszus. G.P. 327,509, 23.3.16.

BORON nitride is changed by carbon at the temperature of the electric arc to a coherent mass. Carbon monoxide, carbon and ammonia, or hydrogen and carbon bisulphide can also be used as reducing agent. For this reaction a temperature of 2000° C. is required; on heating to a considerably higher temperature the carbide melts and gives a product suitable for use as an abrasive.—C. I.

*Argon and other rare gases of the atmosphere; Purification of —.* E. Barbet et Fils et Cie. E.P. 129,939, 15.7.19. Conv., 17.7.18.

IN the process described in E.P. 131,321 (J., 1919, 766 A), the liquid argon, containing a low percentage of nitrogen, which is drawn off from the air rectifier, passes into another plate rectifying column, which is also heated by a worm containing cold gaseous nitrogen under a pressure of 4–5 kg. per sq. cm. Part of the oxygen which is separated is

drawn off at the lower portion, while the remainder condenses and vaporises the argon and nitrogen in the upper portion of the rectifier. These vapours pass into a condenser immersed in liquefied nitrogen drawn from the top of the air rectifier, and thence to a separator, from which liquid argon is returned to the top plates of the argon rectifying column, while uncondensed vapours are passed into the air rectifying column at a point above that where the liquid argon is drawn off. The liquid nitrogen from the heating worm is mixed with that from the worm in the air rectifier and passed to the upper plates of the latter. Neon and helium, together with some hydrogen, which are mixed with the nitrogen from the air rectifier, are first compressed and cooled, and then passed into a rectifier where the nitrogen is collected at the bottom, while the vapours of neon, helium, and hydrogen escaping at the top pass through a coil immersed in nitrogen drawn from the lower part of the column. Neon is thus liquefied and is drawn off, and the hydrogen escapes with the helium in a gaseous state and is separated from it by passing over wood charcoal or by the eudiometric method. Xenon and krypton, which are drawn off from the bottom of the air rectifier, are separated from oxygen in a column from which the latter is separated in the upper portion by heating with the krypton vapours, and then purified by liquid nitrogen; the xenon and krypton are separated at the top and bottom, respectively, of the lower half of the column, which is heated by a worm through which air passes at normal temperature.—W. J. W.

*Hydrogen and mixtures of hydrogen and nitrogen; Production of — [from gases from blast furnaces and electric smelting furnaces].* C. Toniolo, and Officine Elettrochim. Rossi. E.P. 152,975, 24.12.19. Conv., 21.10.19.

GASES from blast furnaces and electric smelting furnaces are treated with steam in presence of a catalyst, such as iron oxide with or without an alkali metal oxide, chromium oxide, or an alkaline-earth oxide, and the hydrogen, or hydrogen and nitrogen mixture, is freed from carbon monoxide, carbon dioxide, and steam by suitable methods. The gases so produced may be employed for the hydrogenation of fats, synthetic manufacture of ammonia, or preparation of explosive mixtures for internal combustion engines, supplementary hydrogen or nitrogen from another source being added, if desired.—W. J. W.

*Hydrogen; Manufacture of — by means of silicon and its alloys.* G. F. Jaubert. U.S.P. 1,366,185, 18.1.21. Appl., 29.7.20.

SUFFICIENT water is added to powdered ferrosilicon to decompose the contained phosphide and produce a pasty mass, and the latter is added to a solution of caustic alkali in a hydrogen generator.—W. E. F. P.

*Hydrogen and carbonates; Process for the simultaneous manufacture of compressed —.* J. Billwiller. G.P. 328,637, 3.10.16.

PEAT, lignite, or the like, is oxidised with solutions or suspensions of basic substances at about 300° C., under pressure, either alone or in the presence of a catalyst.—C. I.

*Sulphur; Method of and apparatus for separating — from sulphur ores.* N. E. Katz, Assr. to H. M. Little. U.S.P. 1,365,922, 18.1.21. Appl., 24.6.19.

THE ore is heated with water in an autoclave until the sulphur is melted by the steam generated.—W. E. F. P.

*Graphite; Manufacture of — in the electric furnace.* C. Conradty. G.P. 327,911, 2.11.17.

THE conversion of coal, coke, or anthracite into graphite proceeds more readily at a pressure less than atmospheric. Such a pressure is obtained in practice by enclosing the furnace in a strong sheet iron vessel and exhausting the latter.—C. I.

*Sulphuryl chloride; Manufacture of —.* T. H. Durrans, Assr. to A. Boake, Roberts and Co. U.S.P. 1,364,738, 4.1.21. Appl., 28.5.18.

SEE E.P. 124,542 of 1918; J., 1919, 361 A.

*Ammonium sulphate; Process of making —. Process of preparing pure ammonia from ammonia-containing gases.* G. N. Vis. U.S.P. 1,366,302—3, 18.1.21. Appl., 3.12.19.

SEE E.P. 136,833—4 of 1918; J., 1919, 516 A, 741 A.

*Hydrogen and carbon monoxide; Treatment or purification of mixtures of — for the separation therefrom of the latter.* J. Harger and H. Terrey. U.S.P. 1,366,176, 18.1.21. Appl., 18.4.18.

SEE E.P. 127,609 of 1917; J., 1919, 577 A.

*Drying salts.* E.P. 156,963. See I.

*Refractory oxides.* U.S.P. 1,352,387—8. See VIII.

## VIII.—GLASS; CERAMICS.

*Porcelain; Transparency and refractoriness of —.* T. Hertwig. Sprechsaal, 1921, 54, 11—12.

FIVE porcelain mixtures with a firing temperature corresponding to Seger cone 9 (1230° C.) were prepared from Zettlitz kaolin, Rörstrand felspar, Pleystein quartz, calcite, and zinc oxide. The first, corresponding to the formula, (0.65K<sub>2</sub>O, 0.35CaO, ZnO).2.8Al<sub>2</sub>O<sub>3</sub>.14SiO<sub>2</sub> (acid ratio 1:1.49) had a shrinkage of 12.7% and was transparent. Small dishes with flat lids of normal thickness were cast quite satisfactorily, but very thin lids sank slightly. Two mixtures of the formulae (0.8K<sub>2</sub>O, 0.2CaO, ZnO).2.8Al<sub>2</sub>O<sub>3</sub>.17SiO<sub>2</sub> (acid ratio 1:1.8) and (0.8K<sub>2</sub>O, 0.2CaO, ZnO).2.8Al<sub>2</sub>O<sub>3</sub>.18SiO<sub>2</sub> (acid ratio 1:1.91) were not refractory, and similar lids made of them collapsed; this is ascribed to the high content of alkali in conjunction with a high acid ratio. A mixture of formula, (0.83K<sub>2</sub>O, 0.17CaO, MgO, FeO).3.59Al<sub>2</sub>O<sub>3</sub>.16.36SiO<sub>2</sub> (acid ratio 1:1.38) was highly transparent and retained its shape completely at Seger cone 14 (1410° C.). A mixture of formula (0.65K<sub>2</sub>O, 0.35CaO, MgO, FeO).3Al<sub>2</sub>O<sub>3</sub>.11SiO<sub>2</sub> (acid ratio 1:1.1) was a biscuit mass, with a good matt appearance but of low transparency. The results confirm Dorfner's statement (J., 1916, 602) that excellent porcelain to be fired at Seger cone 9 can be made, but to ensure the ware retaining its shape perfectly, a high potash content must accompany a low acid ratio, and the quartz content must be kept low or the ware will have a greasy appearance. German porcelains are too rich in almina and quartz and often too low in potash, but if the proportions are altered whilst ignoring the acid ratio the product will not be sufficiently refractory. A glaze suitable for the first mixture mentioned above is composed of grog from the same mixture 22.54%, Rörstrand spar 22.30%, dolomite 9.61%, calcite 7.27%, zinc oxide 2.05%, calcined Zettlitz (Zebisch) kaolin 8.49%, Pleystein quartz 27.74%, corresponding to (0.2K<sub>2</sub>O, 0.2MgO, 0.5CaO, 0.1ZnO).0.5Al<sub>2</sub>O<sub>3</sub>.4SiO<sub>2</sub>. The inclusion of a little grog increases the adhesion of the glaze and the zinc oxide increases its whiteness. No raw clay is needed.—A. B. S.

## PATENTS.

*Reflecting surfaces on glass; Production of — by means of metals in the platinum group.* J. Rheinberg. E.P. 156,472, 16.9.20.

HOMOGENEOUS, structureless mirrors are produced by coating glass with a film of collodion containing a salt of platinum, or of a metal of the platinum group, and submitting the coated glass to the requisite temperature, e.g., 600°–750° C., in a furnace. The necessary temperature is usually lowered about 30° C. by the addition of a small quantity of a lead or bismuth salt to the platinum or other salt.—H. S. H.

*Abrasive stone.* Norton Co., Assees. of J. H. Stean. E.P. 139,502, 25.2.20. Conv., 26.2.19.

AN abrasive stone, which can be used without oil, is composed of a fast-cutting abrasive such as corundum, emery, crystalline alumina, silicon carbide, etc., in very fine powder, with less than 15% of graphite as a non-oily lubricating filler and 5–30% of an organic binding agent such as shellac, resin, bakelite, celluloid, casein, rubber, or rubber substitute. One side of the stone may be composed of abrasive, 80%, and binder, 20%, and the other side of abrasive, 65%, graphite, 10%, binder 25%. The materials are mixed, moulded, heated to 350°–375° F. (about 180°–190° C.) for about 2 hrs. to melt the binder, then subjected to a pressure of about 1 ton per sq. in., and afterwards baked at about 400° F. (200° C.).—A. B. S.

*Abrasive and refractory article and method of producing same.* A. H. Andersen, Assr. to Norton Co. U.S.P. 1,364,849, 4.1.21. Appl., 17.4.19.

A composition for forming ceramic articles comprises refractory or abrasive crystalline grains, a plastic clay, water, and an oily lubricating agent.—H. S. H.

*Refractory materials for furnace linings; Manufacture of —.* A. Rollason. E.P. 156,447, 21.4.20.

CRUSHED dolomite or magnesite is mixed with 5–10% of crushed basic slag and passed through a basic-lined rotary kiln, internally fired to approximately 1800° C. The material leaving the kiln is passed into a closed chamber, where it is annealed and cooled slowly out of contact with the atmosphere, by which treatment its capacity of "knitting together" when mixed with any suitable binding material is considerably increased.—H. S. H.

*Refractory material; Manufacture of —.* J. F. Mollen and W. W. Patnoe, Assrs. to The Dolomite Products Co. U.S.P. 1,365,336, 11.1.21. Appl., 2.2.20.

REFRACTORY material suitable for furnace linings is made by adding a small percentage of common salt to a slurry of raw dolomite, and then passing the mixture through a rotary kiln operated at a temperature sufficiently high to calcine the dolomite and drive off practically all of the salt.—H. S. H.

*Refractory oxide; Method of converting —. Method of shrinking magnesia.* L. E. Saunders, Assr. to Norton Co. U.S.P. (A) 1,352,387 and (B) 1,352,388, 7.9.20. Appl., (A) 15.4.19, (B) 23.7.17. (A) Renewed 17.2.20.

FOR (A) shrinking, agglomerating, sintering, or fusing refractory oxides, or (B) shrinking magnesia, the material is heated locally by the passage of an electric current through, for instance, a thin carbon rod embedded in the charge above the median horizontal line. The carbon burns away, and the charge in the vicinity agglomerates and becomes electrically conductive and then serves to carry the current.

*[Silica] brick and process of making the same.* F. Orth. U.S.P. 1,363,264, 28.12.20. Appl., 29.3.17. Renewed 7.5.20.

A MIXTURE of pure silica (the largest particles passing a 40-mesh screen and the whole graded so as to secure a minimum percentage of voids) with 2–5% of hydrated lime is moulded, treated with steam at a pressure of 125 lb. per sq. in. for 10 hrs., and afterwards fired for 8 days at 2800°–3000° F. (1540° C.—1650° C.).—A. B. S.

*Radium luminous article and method of producing same.* E. O'Hara. U.S.P. 1,364,951, 11.1.21. Appl., 26.12.19.

THE article to be rendered luminous is coated with a paste containing a mixture of a radium luminous material, e.g., radioactive zinc sulphide crystals, and a flux or glaze, and then heated in a muffle furnace so as to melt the glaze.—B. V. S.

*Gas-fire radiants.* E.P. 156,416. See 11c.

*Boron carbide.* G.P. 327,509. See VII.

*Eliminating volatile matter from clay.* U.S.P. 1,363,387. See X.

## IX.—BUILDING MATERIALS.

## PATENTS.

*Insulating and building material.* Fabriks Aktiebolaget Kronsten, Assees. of T. A. Eklund and C. G. Löfveberg. E.P. (A) 140,431, 17.3.20, and (B) 142,111, 21.4.20. Conv., (A) 19.3.19, (B) 24.4.19.

(A) A POROUS insulating material is produced by intimately mixing fresh-water marl with 1–15% of cellulose freed from incrusting substances. Bricks can be moulded or pressed from the wet material. (B) Cellulose-containing material which is not free from incrusting substances (such as moss-litter, peat fibre, kelp, straw, chaff, hemp, flax, mechanical wood pulp, etc.), can replace the cellulose freed from incrusting substances in (A). The results are then improved by the addition of 5–15% of fibrous materials not containing cellulose (e.g., asbestos, slag wool, cow-hair, etc.).—H. S. H.

*Porous stones for thermal insulating purposes; Manufacture of —.* H. J. C. Forrester. From Internationalt Isolations Komp. A./S.-Ikas. E.P. 156,442, 7.4.20.

AN insulating material is obtained by mixing ground "moler" (diatomaceous earth) with a mixture of cork and sawdust, moulding to the required shape, and burning.—H. S. H.

*Asbestos-cement and like cement-mixtures or compounds; Method of and means to cure, colour, or otherwise treat sheets or other articles made of —.* W. Pattison. E.P. 155,883, 11.9.19.

ARTICLES made of asbestos-cement and the like are cured and/or coloured by placing them in a water-tight chamber in which they are subjected successively to various treatments, e.g., heated by means of steam pipes, treated with moist air, neutralising liquids, colouring fluids, etc. The liquids are circulated in a series of similar chambers which are used simultaneously, but the articles remain in the chamber in which they are placed until their treatment is finished.—A. B. S.

*Coating process [for cement].* L. A. and A. J. Sanders. U.S.P. 1,364,587, 4.1.21. Appl., 10.12.15.

SURFACES of calcareous cements of the nature of hydraulic cement, and having an alkaline reaction because of the lime present, are treated with an aqueous solution of a neutral salt of a heavy metal forming coloured oxides and containing an acid not

decomposed by the lime of the surface. A deposit of a coloured metal oxide is formed in the surface layer by interaction between the lime and the acid contained in the salt.—H. S. H.

*Slag.* E.P. 150,033. See IIa.

*Peat.* E.P. 155,895. See IIa.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Iron ores; Decomposition of oxide* — [for analysis]. F. Leteur. Ann. Chim. Analyt., 1921, 3, 16—19.

IRON oxides which are difficult to obtain in solution may be dissolved readily by heating them in a closed flask at 60°–70° C. with concentrated hydrochloric acid, particularly if the latter has been saturated with hydrogen chloride just before use. The pressure developed is not great, and with reasonably strong flasks there is but little risk of breakage.

—W. P. S.

*Graphite; Solubility of* — in molten iron. R. Ruer and J. Biren. Z. anorg. Chem., 1920, 113, 98—112.

THE solubility of graphite in molten Swedish iron increases as the temperature is raised from 1152° C. to 2700° C. The solubility curve is linear from 1152° to 1700° C.; it then bends away from the concentration axis, slightly at first and then more strongly as the temperature increases. The concentration of the graphite eutectic at 1152° C. is 4.25% carbon, and that of the cementite eutectic at 1145° C. is 4.30% carbon. (Cf. J.C.S., Mar.)—J. F. S.

*Aluminium; Purification and testing of* —. F. and W. Mylius. Z. anorg. Chem., 1920, 114, 27—64.

WHEN technically pure aluminium (Al 99.58%, Fe 0.18%, Si 0.22%, C 0.02%) was allowed to cool slowly, the portion first crystallising was purer than that which crystallised later, in which the iron and silicon became concentrated. From highly impure aluminium, however, impure crystals of high-melting alloys separated first. In the neighbourhood of its melting point technically pure aluminium becomes brittle, and can be granulated by stirring the metal during crystallisation. Such granulated metal can be almost entirely freed from iron by repeated extraction with dilute hydrochloric acid, which dissolves the impure "cement" material from the purer aluminium crystals. It is best to re-melt and re-granulate the metal between successive extractions. After four such treatments, from the above 99.58% material, an aluminium was prepared, in 50% yield, containing Al 99.9%, Fe 0.02%, Si 0.2%, C 0.01%. Purification to about the same extent was also effected by partially melting the aluminium in a reducing atmosphere and pouring off the lower melting portion from the purer aluminium crystals. The yield of purified metal by this process was low. With the object of devising a test which would indicate the probable behaviour of objects of technical aluminium under atmospheric influences, experiments were made on the action of solvents and oxidising agents on aluminium of different degrees of purity, in the form of castings, foil, or wire. A test was devised with a solution containing 2.0% of hydrogen peroxide and 1% of sodium chloride. The extent of attack by this reagent, as measured by the loss of weight of the sample after immersion for several days under standard conditions, diminishes, in the case of wire, as the purity of the metal is increased. The amount of attack is also much lessened by first removing the effect of cold-work by heating the metal for some time at 500° C. Another test which gave

similar results, consisted in treating the sample to be tested in a test-tube with 10% hydrochloric acid and plotting the time-temperature curve. The rate of rise of temperature to a maximum was found to be a function of the physical and chemical properties of the aluminium. The most highly purified aluminium was scarcely attacked by 20% hydrochloric acid.—E. H. R.

*Nolly's electric apparatus* [for the determination of carbon in metals]; Modifications of —. Bedin. Ann. Chim. Analyt., 1921, 3, 15—16. (Cf. J., 1911, 688, 1216.)

THE modifications suggested are an adjustable resistance, a two-way tap on the vacuum flask, and the use of bismuth tetroxide in place of lead peroxide as oxidising agent. Much practice is required before trustworthy results can be obtained with the apparatus; concordant results cannot be obtained when the carbon content of a metal exceeds 3.5%.

—W. P. S.

*Iron oxides and carbon monoxide.* Chandron. See VII.

*Basic slag.* (1) Jones. (2) Bainbridge. (3) Stead and Jackson. (4) Robertson. See XVI.

## PATENTS.

*Cast iron; Manufacture of soft* —. T. Levoz. E.P. 140,756, 17.2.20. Conv., 27.3.19.

A MIXTURE of hæmatite irons or hæmatite irons and scrap is melted in a converter with a surface blast of air until silicon is completely removed, then transferred to a furnace and heated to not exceeding 1550° C., after which the desired combined carbon content is obtained by the addition of reducers.—J. W. D.

*Copper from nickel; Process for electrolytically separating* —. P. Goldberg. E.P. 145,600, 29.6.20. Conv., 9.7.18.

ANODES containing copper and nickel are treated electrolytically in hydrochloric acid or in an acid solution of a chloride in such a manner that the quantity of copper deposited at the cathode and the quantity dissolved at the anode are equal, the copper content of the solution being kept uniform and the nickel remaining in solution.—J. W. D.

*Magnetic separators.* F. Krupp A.-G. Grusonwerk. E.P. 147,546, 18.6.20. Conv., 11.7.19.

A MAGNETIC separator of the revolving inductor ring type is mounted in such a manner that the whole apparatus, including the feeding device, the magnets, and the means for carrying away the separated material, is capable of being tilted about its driving shaft, without any relative displacement of the several parts, and without interfering with the driving gear.—A. R. P.

*Welding of metal objects* [; Electric —]. C. F. Jacobs. E.P. 156,376, 12.11.19.

THE objects to be welded are immersed in a bath of molten flux, which serves to convey the welding current. The current preferably passes also through one or both of the objects to be welded, and one or both of them are preferably removed from the circuit before the welding process is complete.

—J. S. G. T.

*Electric arc welding and like operations and electrodes therefor.* C. J. Holslag. E.P. (A) 156,280 and (a) 156,476, 30.6.19.

(A) A TWIN or multiple electrode for arc welding etc., comprises two or more metal electrodes arranged adjacent to, but insulated from, each other, so that two closely adjacent arcs are produced having a common puddle of molten metal. The



electrodes are held together by a flux coating, wholly or partly surrounding them, and may be arranged in one or more planes. (b) A number of electrodes conveying currents of different phases are employed to produce separate adjacent arcs, after the manner described under (a).—J. S. G. T.

*Arc-welding electrode.* P. H. Brace, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,363,636, 28.12.20. Appl., 12.5.19.

An arc-welding electrode comprises a sheath of welding metal enclosing a core of an alkaline-earth metal.—J. W. D.

*Copper cable tubes and other metal articles; Apparatus for the manufacture of — by electro-deposition.* S. O. Cowper-Coles. E.P. 156,441, 26.3.20.

APPARATUS for the manufacture of copper cable tubes etc. by electro-deposition comprises a vat having a central partition, rotary devices for rapidly circulating the electrolyte, and a number of cathode matrices, which are oscillated and moved up and down in the electrolyte in which the anodes are suspended. The circulating devices consist of a set of blades disposed vertically, united at their ends by rings, and secured at their centres to a spider disc mounted on a vertical shaft, the whole being mounted in a chamber having a hole at the bottom and a number of smaller holes at the top. Means are provided for continuously drawing off the electrolyte, filtering it, and returning it to the vat. —J. S. G. T.

*Metals; Method of treating — [in electric furnaces].* R. H. Bulley. U.S.P. 1,347,838, 27.7.20. Appl., 24.4.16. Renewed 5.8.19.

THE metal is subjected to the influence of a relatively high voltage electric arc in an electric circuit of a given kilowatt load, and later for the final refining of the metal, the voltage in the circuit is reduced whilst retaining the load. In the latter stages the arc is shorter, and hence there is less destructive action on the roof of the furnace. —J. W. D.

*Ore-concentrating apparatus.* B. H. Dosenbach; E. M. Dosenbach, extris. U.S.P. 1,354,031, 28.9.20. Appl., 28.1.18.

AIR is forced through a chamber containing one of the usual modifying agents employed in flotation processes, e.g., tar oil, alcohols, naphthylamine, nitronaphthalene, naphthol, etc., and the mixture of air and vapour is introduced, under regulated pressure, into ore pulp contained in a flotation cell of the Callow type.

*Ore-concentration process.* W. A. Scott. U.S.P. 1,365,281, 11.1.21. Appl., 8.7.19.

A FREELY flowing ore pulp is treated so as to produce therein bubbles containing a substance capable of reacting chemically with a substance present in the pulp to form a flotation agent. The bubbles with adhering solids are then separated from the remainder of the pulp.

*Flotation of minerals.* C. L. Perkins, Assr. to Metals Recovery Co. U.S.P. (a) 1,364,304, (s) 1,364,305, (o) 1,364,306, and (n) 1,364,353, 4.1.21. Appl., (a) (s) (o) 21.7.19, (n) 13.7.20.

THE mineral pulp is treated with a small amount of (a) a non-oleaginous organic mineral-collecting agent which is substantially non-frothing, (s) an organic nitrogen compound containing two nitrogen atoms joined to each other, or (o) a hydrazine compound, and the resulting mixture is subjected to a froth flotation operation. (n) The pulp is treated with a small amount of a substantially non-frothing, non-oleaginous organic mineral-collecting agent,

then with a substance having good frothing properties, and the resulting mixture is subjected to flotation.—A. R. P.

*Flotation of minerals.* C. L. Perkins and R. E. Sayre, Assrs. to Metals Recovery Co. U.S.P. (a) 1,364,307, (s) 1,364,308, and (o) 1,364,359, 4.1.21. Appl., (a) 11.11.19, (s) 25.3.19, (o) 13.7.20.

A SMALL amount of (a) an organic nitrogen-sulphur compound, or (s) a thionrea compound, is added to the mineral pulp, which is then subjected to a flotation. (o) As a mineral-collecting agent a small amount of a non-frothing, non-oleaginous organic nitrogen-sulphur compound is added to the pulp, which is then treated with a substance having good frothing properties, and the mixture put through the flotation process.—A. R. P.

*Alloy; Metal —.* J. E. Holder. U.S.P. 1,359,064, 16.11.20. Appl., 2.6.20.

AN alloy of lead 30—50, copper 47—68, nickel 1—5, arsenic 0.5—1.5, manganese 2.5, phosphorus 0.5, and iron 0.5 pts.—J. W. D.

*Alloys; Process of producing metallic —.* J. P. Arend. U.S.P. 1,363,382, 28.12.20. Appl., 21.3.18. Renewed 28.8.20.

TO obtain alloys free from oxygen the material is treated during the melting-down operation with a reducing slag containing alkali manganosilicates. —J. W. D.

*Zinc alloy.* A. Tedesco, Assr. to Soc. Anon. Stabilimenti Biak. U.S.P. 1,364,654, 4.1.21. Appl., 11.4.19.

AN alloy containing Zn 85%, Cu 2—8%, Al 2.5—7.1%, Ni 0.5—8%, together with small quantities of iron and manganese.—J. W. D.

*Alloy.* A. W. Clement, Assr. to The Cleveland Brass Manufacturing Co. U.S.P. 1,365,091, 11.1.21. Appl., 12.3.17.

AN alloy of iron and chromium substantially free from carbon, oxides, and impurities is produced either by reducing a combined ore of iron and chromium, followed by decarburising the reduced metal by treatment with oxides of one or more of the component metals, or by decarburising an alloy of iron, chromium, and carbon. The resulting alloy is deoxidised by fluxing with calcium carbide. —J. W. D.

*Aluminium alloy, and method of making same.* A. De Lavandeyra. U.S.P. 1,365,173, 11.1.21. Appl., 7.8.19.

THE alloy contains aluminium and smaller amounts of manganese, magnesium, copper, and chromium. —A. J. H.

*Alloy.* H. S. Foote, Assr. to Standard Chemical Co. U.S.P. 1,366,254, 18.1.21. Appl., 29.9.20.

AN alloy of steel with uranium, silicon, and nickel. —J. W. D.

*Metal-bearing ores or materials; Treatment of — and recovery of the valuable or desired constituents thereof.* E. A. Ashcroft. E.P. 156,866, 30.9.19.

THE ore, suspended in a mobile fused melt, is treated in a converter with chlorine or a gas yielding chlorine, thus converting the metals into chlorides. Iron or manganese is converted into oxide by precipitating with magnesia, lime, or alkali oxide or carbonate, removing the insoluble gangue matter and the precipitated oxide of iron or manganese by filtration, fractionally precipitating the metals which it is desired to recover (for example, silver, lead, and zinc) by means of an alloy of magnesium, calcium, sodium, or potassium



with lead or zinc, and electrolysing the remaining chlorides with cathodes of the heavier metals so as to re-form the alloys for re-use and produce a cyclic process.—J. W. D.

*Ores, minerals, and clays; Process for eliminating volatile matter and gases from —.* S. L. Boggs. U.S.P. 1,363,387, 28.12.20. Appl., 2.8.20.

THE material is roasted, crushed, and pulverised, and then agitated in a mixture of alum and water.—W. J. W.

*Casting freely-oxidisable metals.* H. E. Bakken, Assr. to Aluminium Co. of America. U.S.P. 1,363,384, 28.12.20. Appl., 15.1.20.

THE metal is cast in a mould made from sand which is free from water and which is rendered mouldable by being mixed with a non-aqueous liquid.—J. W. D.

*Ores; Method of treating —.* G. Moore. U.S.P. 1,364,573, 4.1.21. Appl., 14.1.19. Renewed 20.7.20.

SULPHIDE ores are subjected in a closed container to the action of sulphuric acid and ozonised air under the influence of heat, the evolved sulphur dioxide being converted into sulphuric acid which is then returned to the container.—J. W. D.

*Ores; Process of and apparatus for reducing and smelting —.* J. Lund. U.S.P. 1,365,113, 11.1.21. Appl., 26.6.16.

THE apparatus comprises a furnace having a reducing chamber, means for feeding ore and fuel to this chamber, also means for supplying compressed air to, and preventing free escape of gas from, the chamber, a smelting chamber communicating with the reducing chamber, and so constructed that a liquid resistance to compressed gases can be maintained between the two chambers.—J. W. D.

*Nickel and cobalt; Process of separating —.* M. J. Udy and O. C. Ralston, Assrs. to Hooker Electrochem. Co. U.S.P. 1,365,358, 11.1.21. Appl., 3.7.19.

COBALT is precipitated from mixed nickel cobalt solutions by simultaneously subjecting the solution to the action of chlorine and an alkaline-earth carbonate at a temperature below that at which material precipitation of nickel occurs.—J. W. D.

*Slag-pocket floor.* S. G. Wörton. U.S.P. 1,365,436, 11.1.21. Appl., 3.2.17.

A SLAG-POCKET is interposed between the regenerator of a regenerative furnace and a flue opening into the interior of the furnace. The slag-pocket has a removable hollow metal floor supported on a refractory bottom, with means for circulating a cooling medium within the hollow floor.—J. W. D.

*Surface-alloyed metal.* F. C. Kelley, Assr. to General Electric Co. U.S.P. 1,365,499, 11.1.21. Appl., 23.10.19.

To provide a metal with a surface-alloy of chromium the metal is heated in a non-oxidising atmosphere in contact with powdered metallic chromium to a temperature of at least 1200° C., but below the melting-point of the metal.—J. W. D.

*Metal from ore; Method of obtaining —.* S. G. Musser. U.S.P. 1,365,844, 18.1.21. Appl., 19.1.18. Renewed 13.5.20.

METAL is obtained from ore by means of a centrifugal amalgamator, in which a hollow rotating column of ore is caused by centrifugal action to move through and in contact with a hollow body of mercury. The tailings are discharged in a

direction opposite to the direction of rotation, so as to prevent close packing of the ore in the column.—J. W. D.

*Magnetic qualities of magnetisable material; Process of improving the —.* Metropolitan-Vickers Electrical Co., Ltd., Asses. of T. D. Yensen. E.P. 141,348, 6.4.20. Conv., 4.4.19.

SEE U.S.P. 1,358,810 of 1920; J., 1921, 17 A.

*Tin-scruff produced in the process of making tin andterne plates; Treating —.* G. H. Clegg. U.S.P. 1,365,456, 11.1.21. Appl., 14.6.20.

SEE E.P. 151,374 of 1919; J., 1920, 753 A.

*Hydrogen, or hydrogen and nitrogen.* E.P. 152,975. See VII.

## XI.—ELECTRO-CHEMISTRY.

*Electrical conductivity of electrolytes at temperatures up to 1600° C.; Accurate measurement of the —.* F. M. Jaeger and B. Kapma. Z. anorg. Chem., 1920, 113, 27–58.

THE electrical conductivity of molten electrolytes may be measured in iridium-free platinum vessels heated in a tubular electric furnace at temperatures up to 1600° C. (Cf. J.C.S., March.)—J. F. S.

*Arc process of nitrogen fixation.* Hagemann. See VII.

*Soya bean oil.* Dall'Acqua. See XII.

*Electrolytic reduction of menthone.* Matsui and Shimizu. See XX.

### PATENTS.

*Electric [crucible] furnace.* E. Assié. E.P. 141,352, 6.4.20. Conv., 9.8.17.

AN electric crucible furnace for making refractory products comprises a hearth and outer refractory casing of beaten graphite, an inner casing of agglomerated material such as calcined magnesite, dehydrated bauxite, carbon, or corundum dust, which is a conductor only at a high temperature, and a movable electrode, the whole being enclosed within a sheet metal casing.—J. S. G. T.

*Electrodes of electrolytic cells; Protecting varnish for —.* T. A. Edison. U.S.P. 1,364,359, 4.1.21. Appl., 29.9.19.

A VARNISH for protecting electrodes of electrolytic cells is composed of a coumarone or paracoumarone resin.—J. S. G. T.

*Electrolytic apparatus.* W. E. Greenawalt. U.S.P. 1,365,032–4, 11.1.21. Appl., (a) 29.4.18, (b) 10.2.19, and (c) 13.6.14. (c) Renewed, 5.4.19.

(A) THE electrodes are so arranged in an electrolytic chamber that the electrolyte takes a sinuous path as it flows through the chamber. A gas is also passed through the chamber, and means are provided for spraying the liquid into the gas at the crests of the sinuous path. (a) An electrode consists of an interior support composed of sections bolted together internally with rods, and having metal sheets attached to the faces of the support. (c) A portion of the electrolyte is withdrawn from an electrolytic chamber and passed into a separate vessel in which a "finely divided sulphide reagent" is maintained in suspension in the electrolyte by agitation with an atomised gaseous reagent. The gas is atomised by leading it into the liquid below a rotating perforated agitator submerged in the liquid, and, after treatment with gas, the liquid is separated from insoluble matter and returned to the electrolytic chamber.—L. A. C.

**Electrolytic cell.** L. E. Ward, Assr. to The Dow Chemical Co. U.S.P. 1,365,875, 18.1.21. Appl., 13.12.18. \*

THE space between two plates in an electrolytic cell is closed laterally so as to form a chamber for the electrolyte. This chamber is divided into compartments by a metallic screen covered by a diaphragm. Carbon electrodes project through the plates, one set of electrodes being connected with the screen. Means are provided for adjusting the height of electrolyte in the compartments.—J. S. G. T.

**Electrical furnaces; Method for the heating of material or the performance of chemical processes in —, with apparatus intended therefor.** Aktiebolaget Kvalveindustri. E.P. 141,656, 16.7.19. Conv., 12.4.19.

SEE U.S.P. 1,342,809 of 1920; J., 1920, 550 A.

**Electrically-heated crucible, melting-pot, and the like.** C. W. Speirs, Assr. to Morgan Crucible Co., Ltd. U.S.P. 1,366,135, 18.1.21. Appl., 2.6.19.

SEE E.P. 129,407 of 1918; J., 1919, 685 A.

See also pages (A) 135, *Electro-osmotic separation* (E.P. 144,710), 143, *Insulating material* (E.P. 131,907), 149, *Refractory oxides* (U.S.P. 1,352,337—8), 150, *Separating copper from nickel* (E.P. 145,600); *Electric welding* (E.P. 156,280, 156,376, 156,476), 151, *Arc-welding electrode* (U.S.P. 1,363,636); *Copper cable tubes* (E.P. 156,441).

## XII.—FATS; OILS; WAXES.

**Soya bean oil; Characterisation of — electrically.** G. Dall'Acqua. Giorn. Chim. Ind. Applic., 1920, 1, 48—53.

VARIOUS characters of soya bean, maize, and grape-seed oils are compared. Comparatively small differences are shown by the molecular weights in freezing benzene, or by refractive indices or their temperature coefficients, of the oils themselves or their fatty acids. Discharge of an electroscope of the Elster and Geitel type, however, occupies only a fraction of a second when taking place through soya bean oil and from 15 to 100 seconds with various other oils or their fatty acids; in the case of a very old sample of soya bean oil, which was profoundly changed, 1.6 seconds was required.—T. H. P.

**Lemon-seed oil.** Bertolo. Giorn. Chim. Ind. Applic., 1920, 1, 54—55.

LEMON-SEEDS occur in large quantities in the so-called *pastazzo*, which forms the residue of the citrus industry. When pressed the material yields little oil, but extraction with carbon bisulphide, petroleum spirit, or benzene gives 30—35% of oil according to the degree of maturation of the seeds. The expressed oil is clear but the extracted oil is turbid and gradually deposits considerable sediment consisting largely of solid, saponifiable substances; the oil retains a pronounced odour of lemons and a somewhat bitter taste; it has a dark yellow colour and a green fluorescence, which is not removed by repeated washing with hot water or dilute sulphuric acid. Its characters are as follows: sp. gr. at 22°, 0.9160 for the expressed, and 0.9180 for the extracted and washed oil; freezing point, -5° to -6°; Zeiss butyrefractometer reading, 74 at 17° C. and 70 at 25° C.; Maumené value, 78; critical temperature of solution in acetic acid (Valenta test), 108°; oxygen absorbed (Livache's method), 5.4%; m.p. of fatty acids, 41°; solidif. p. of fatty acids, 35—38° C.; free fatty acids (as oleic acid), 2.82%; saponif. value, 190—191; iodine value, 103—108; Hehner value, 94. The oil is a semi-drying oil. It does not give Halphen's reaction.—T. H. P.

**Fatty oils from Reptilia and Amphibia.** M. Tsujimoto and S. Kobayashi. Kōgyō Kwagaku Zasshi (J. Chem. Ind. Tokyo), 1920, 23, 1099—1109.

THE paper contains the results of an examination of the oils from certain *Reptilia* and *Amphibia*, viz., blubber oil from "leather turtle," Japanese "Osagame"; oil from "Akaumigame," red turtle, *Caretta olivacea*, Escholtz; blubber oil and liver oil from "Migame," *Clemmys japonica*, Temminck and Schlegel; oil from a Japanese viper, "Mamushi," *Agkistrodon blomhoffii* (Boie); oil from a snake called "Yamakagashi," *Natrix tigrina* (Boie), a harmless species common in Japan; "Aodaisho" oil from *Elaphe climacophora* (Boie), the largest harmless snake found in Japan; python oil from *Python reticulatus*, Schin.; oil from a giant lizard (*Varanus*); toad oil from "Hikigaeru," *Bufo bufo japonicus* (Schlegel); and giant salamander oil from *Megalobatrachus japonicus* (Temm.).—K. K.

**Emulsions; Three-phase —.** H. Bechhold, L. Dede, and L. Reiner. Kolloid Zeits., 1921, 28, 6—19.

PAIRS of immiscible liquids such as benzene-water and petroleum-water may be emulsified by shaking with clay, zinc-dust, or yeast. Emulsion formation depends on the quantity of the powder added, the size of the particles, and on the presence of certain other substances termed emulsion promoters. The emulsifying power of the powder (emulsion former) increases to a maximum with decreasing size of the particles and then decreases. Benzene and water are not emulsified by shaking with clay alone, but the addition of a few drops of such substances as pyridine, acetic acid, ethyl alcohol, sodium hydroxide, or sodium nitrate immediately causes emulsification. (Cf. J.C.S. March.)—J. F. S.

**Oxidation of lignite products.** Schneider. See 111.

**Lard.** Kerr. See XIXA.

**Hydrogenation.** Willstätter and Waldschmidt-Leitz. See XX.

## PATENTS.

**Fat or oil; Extraction of — from raw materials.** H. Bollmann. E.P. 156,905, 14.10.19.

SEE G.P. 303,846 and 322,446; J., 1920, 459 A, 728 A.

**Purifying oils.** U.S.P. 1,365,894. See IIA.

**Tyre filler.** E.P. 140,781. See XIV.

**Margarine.** E.P. 156,000. See XIXA.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Resins; Constituents of —.** VII. *Lubanyl benzoate from Siamese benzoin.* I. A. Zinke and J. Dzimal. Monatsh., 1920, 41, 423—441.

INVESTIGATION of the chemical transformations undergone by lubanyl benzoate (J., 1915, 681) indicates for this compound the constitution  $(4,HO)(3,CH_3)C_6H_4(1)C_6H_4O.CO.C_6H_5$ . Further, since the benzoate yields eugenol or isoeugenol on reduction, the  $C_6H_5$  residue must represent a direct chain and must contain the double linking; it is possible that this compound is the benzoate corresponding with coniferyl alcohol. (Cf. J.C.S., March.)—T. H. P.

## PATENTS.

**Lamp black; Process for the production of —.** J. M. Gerard and H. J. Masson, jun. U.S.P. 1,364,273, 4.1.21. Appl., 23.12.18. Renewed 29.5.20.

A GASEOUS or vaporised carbon compound is decom-

posed with formation of free carbon by contact with an inert gas at high temperature or by the heat developed in an exothermic gas reaction. The reaction products are rapidly removed from the heated zone in order to ensure a deep black colour in the carbon; further reaction is prevented by spraying with water, whilst the temperature is maintained above 100° C., and the carbon is collected by electrical precipitation.—D. F. T.

**Resins; Production of** — [from naphtha]. S. P. Miller and F. H. Rhodes, Assrs. to The Barrett Co. U.S.P. 1,365,423, 11.1.21. Appl., 28.8.19.

The solution of polymerised substances obtained in the production of resins from the polymerisable constituents of naphtha is washed with a solution of an acid before the succeeding operations.—D. F. T.

**Japan.** H. Chislet, Assr. to General Electric Co. U.S.P. 1,357,688, 2.11.20. Appl., 11.7.19.

SEE E.P. 155,427 of 1919; J., 1921, 91 A.

**Varnish for aircraft constructions; Dope or** —. S. E. Groves. U.S.P. 1,366,256, 18.1.21. Appl., 2.8.18.

SEE E.P. 128,974 of 1917; J., 1919, 648 A.

**Bituminous material.** U.S.P. 1,340,855. See III.

**Resins.** G.P. 326,729. See III.

**Cellulose acetate solutions.** E.P. 145,511. See V.

**Varnish for electrodes.** U.S.P. 1,364,359. See XI.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

##### PATENTS.

**Vulcanisation of natural or artificial rubber or rubber-like substances; Process of** —. C. R. de Long and W. N. Watson. U.S.P. 1,364,732, 4.1.21. Appl., 20.7.20.

**DICHLOROANILINE** is introduced into the rubber mixture prior to vulcanisation.—D. F. T.

(A) Sulphur-terpene compound. (B) Waterproofed fabric. (C) Vulcanised article and process of producing the same. (D) Article and process of bonding metal and vulcanised rubber. W. B. Pratt, Assr. to E. H. Clapp Rubber Co. U.S.P. 1,349,909—12, 17.8.20. Appl., (A) 3.10.19, (B) 8.1.19, (C) (D) 5.5.20. (E) Renewed 3.5.20.

(A) TURPENTINE oil and sulphur are heated together above 150° C. at atmospheric pressure, whereby hydrogen sulphide is evolved and a product containing 30–50% S is obtained as a thick viscous liquid or a black solid, suitable for use in rubber mixings. A solution of the solid product in toluene or xylene may be used for impregnating cotton or the like. (a) Fabric impregnated with a sulphur-terpene compound (cf. A) is used for the manufacture of rubber-proofed goods. (c) Fibrous material in strip or sheet form is treated with the sulphur-terpene compound, then coated with a rubber composition and vulcanised. (n) Metal is coated with the sulphur-terpene compound, a layer of vulcanised rubber is then applied, and the whole is subjected to heat and pressure.

**Tyre filler.** F. A. Hager. E.P. 140,781, 23.3.20. Conv., 11.4.18.

A MIXTURE of soya bean oil (24 lb.), magnesium oxide (17½ oz.), Venetian red (3 oz.), and sulphur chloride (6 lb.) is poured into moulds. After solidification it is introduced into the cover of a pneumatic tyre in place of the usual tube of compressed air.—D. F. T.

#### XV.—LEATHER; BONE; HORN; GLUE.

**Pelt; Swelling of** —. G. Graessner. Collegium, 1920, 353–359, 405–416, 456–464, 512–518.

DIFFERENT organic acids behave similarly in regard to their action on pelt swelling; the maximum swelling is only attained after some hours, and the optimum concentration is different for different acids. Monochloroacetic acid gives a maximum swelling with the lowest concentration, then follow oxalic, lactic, formic, and acetic acids. The order of increasing times of attaining maximum swelling is:—Oxalic, lactic, acetic, monochloroacetic, formic acid. The greatest swelling in unit time and per unit concentration is given by monochloroacetic acid, and comparison of the dissociation constants shows no apparent connexion between these and the swelling in unit time and per unit concentration. The velocity of swelling is not influenced by the dissociation of the acids. The influence of salts on swelling is not very definite. Sulphates repress swelling very rapidly; the action of nitrates is more gradual, but ultimately is greater than that of sulphates at the highest concentrations. Dissociation does not explain the repressing effects of salts. This repression is not proportional to the concentration of the salt solutions used, hence it is not a purely osmotic effect. Cuttings of pelt treated with dilute hydrochloric acid solutions with varying additions of sugar solution showed increased swelling and acid adsorption with increased addition of sugar. The sugar did not repress the swelling, confirming the view that the effect of neutral salts on swollen pelt is not an osmotic process. The amount of organic acids absorbed was fairly constant and remained uninfluenced by the addition of salts; it was about 1.15 c.c. of N/1 acid for 2 g. of pelt. This result indicates that the absorption is a chemical process with the formation of salts of hide substance. The average water content of pelt being 58%, 100 g. of dry hide substance requires 5 g. of hydrochloric acid, which agrees with the formula  $C_{12}H_{19}O_{11}N_3.HCl$  for collagen hydrochloride and indicates a free amino-group in collagen.—D. W.

**Synthetic tannins; Tanning and chemical properties of the sulphonic groups in** —. W. Moeller. Collegium, 1920, 520–536.

STRONG solutions of synthetic tannins attack hide substance and leather. Dilute solutions, therefore, should be used, and careful after-treatment is necessary to obviate subsequent deterioration. Vegetable tannins, phenol, and gallotannic acid do not attack the hide substance, hence in vegetable tanning the hydrolysis ceases. Tannins may be defined as substances which, either alone or in contact with hide substance, form colloidal solutions, which prevent decomposition of the hide by hydrolysis and fermentation and are irreversibly adsorbed by it. Vegetable tannins diminish the "proteolytic constant," i.e., the quantity of hide substance dissolved as a result of hydrolysis; Neradol D and ND, Ordoval G and 2 G increase it. The addition of 50% of vegetable tannin to synthetic tannin reduces the extent to which the "proteolytic constant" is increased. Sulphuric acid does not increase the constant much, but  $\beta$ -naphthalene-sulphonic acid increases it considerably. The acidity of synthetic tannins diminishes after they have been in contact with hide powder. The free sulphonic acids in the synthetic tannins split up the micellar bands of the hide fibrils into free micells, which are subsequently broken up into protein molecules, and these are further hydrolysed to amino-acids, which combine with the sulphonic acids to form soluble condensation products; hence the diminished yield of leather with even moderate use of synthetic tannin. The use of more synthetic tannin does not result in increased

weight, and leather tanned with it should be well washed to remove excess tannin. Defects arising in leathers tanned with synthetic tannins are not due to free sulphuric acid, but to the sulphonic acids, which are more reactive, causing brittleness and deterioration. Only a very small portion of synthetic tannin acts as true tannin. Similar effects can be obtained by adding free mineral acid or any sulphonic acid to a vegetable tanning material. Synthetic tannins appear to be related to bates, and where a loose leather is not a disadvantage the bating could be shortened and synthetic tannins employed in the tanning process to carry on the bating simultaneously with the tanning.—D. W.

*Basicity of chrome [tanning] liquors; Form of expression for the —.* K. Schorlemmer. Col. legium, 1920, 536—538.

THE usual chrome liquor analysis gives the percentage of  $\text{Cr}_2\text{O}_3$  (a) and of  $\text{SO}_3$  in the sample. The  $\text{SO}_3$  is calculated in terms of  $\text{Cr}_2\text{O}_3$  (b), and  $100b/a$  gives the percentage of  $\text{Cr}_2\text{O}_3$  combined with its full equivalent of acid. The percentage of chromium combined with hydroxyl groups is obtained by deducting this figure from 100, and the resulting figure is an expression of the basicity. The following are the basicities obtained:  $\text{Cr}_2(\text{SO}_4)_3$ , 0;  $\text{Cr}(\text{OH})\text{SO}_3$ , 33.3%;  $\text{Cr}_2(\text{OH})\text{Cl}_2$ , 50.0%;  $\text{Cr}(\text{OH})_3$ , 100.0%.—D. W.

#### PATENTS.

*Patent leather; Process for drying (hardening) —.* C. Heyl. G.P. 327,794, 19.9.15.

THE material is dried in an oven through which a current of dry, warm air passes. The air may be dried by cooling. Moist air hardens the leather in an unsatisfactory manner, and the defect cannot be rectified in the subsequent hardening process.—A. J. H.

*Patent leather; Process for drying — under the influence of ultra-violet light.* C. Heyl. G.P. 323,241, 24.10.19. Addn. to 303,096.

THE leather is dried under the influence of ultra-violet light in an atmosphere of indifferent gases (nitrogen, carbon dioxide, or gaseous products of combustion) containing not more than 2% by vol. of oxygen (10% of air). The temperature is preferably below  $50^\circ\text{C}$ . and the air is as dry as possible. Under these conditions, the amount of ozone formed is negligible.—A. J. H.

*Leather; Manufacture of —.* J. Reerink. G.P. 323,240, 26.9.18.

AFTER treatment with sodim thiosulphate, raw hides are treated with dilute sulphite-cellulose liquors to which may be added water-soluble "tanning oils." The hides are then tanned and prepared in the usual manner, whereby a supple and highly durable leather is obtained.—A. J. H.

*Catgut; Manufacture of —.* C. Braun. G.P. 327,402, 7.8.17.

THE separated intestines are treated, either before or during disinfection, with dilute acids or with solutions of acid salts or salts which have an acid reaction when dissolved in water.—A. J. H.

*Glue, gelatin and the like; Apparatus for the extraction of —.* F. H. Tunnell. U.S.P. 1,364,904, 11.1.21. Appl., 28.6.19.

AN extracting liquid in intimate contact with the glue-yielding material is locally heated so as to set up circulation, whereby the liquid is raised to a point above the said material, diffuses over its surface, percolates through it, and returns to the starting point.—D. W.

*Leather; Process for making — with the assistance of iron salts.* O. Röhm. U.S.P. 1,364,316—7, 4.1.21. Appl., 10.3.17. Renewed 28.6.20.

See E.P. 103,827 of 1917; J., 1918, 14 A.

#### XVI.—SOILS; FERTILISERS.

*Soil solution; Relation of the — to the soil extract.* D. R. Hoagland, J. C. Martin, and G. R. Stewart. J. Agric. Res., 1920, 29, 381—395. (Cf. Hoagland, J., 1918, 252 A.)

FURTHER evidence is given showing that water extracts reflect the principal fluctuations taking place in the soil solution due to season and crop growths as indicated by the freezing-point method. Soil extracts were prepared using 1 pt. of soil to  $\frac{1}{2}$  or 1 pt. of water, which, when concentrated so that they showed the same freezing-point depression as the moist soil, did not alter appreciably in concentration or composition on contact with the soil. It is probable that the ratios between most of the important elements or ions are very similar in concentrated soil extracts and in the soil solution. Suitable soil extracts may be made by extracting 1 pt. of soil with 1 pt. of water, or at the most 5 pts. of water, the time of contact being limited to that necessary for thorough admixture. Analysis of such extracts and determination of freezing-point depressions would probably permit of a determination of the concentration and approximate composition of the soil solution. (Cf. J.C.S., March.)—W. G.

*Soil; Effect of season and crop growth on the physical state of the —.* D. R. Hoagland and J. C. Martin. J. Agric. Res., 1920, 29, 397—404.

THE physical state of certain soil constituents is influenced to a marked degree by the concentration of the soil solution. There is a considerable seasonal variation in the colloidal state of the soil, as determined by turbidity measurements, and a large increase in colloidal matter is noted when the soil solution is depleted as a result of absorption of solutes by the plant.—W. G.

*Azotobacter; Influence of luminous radiations on —.* E. Kayser. Comptes rend., 1921, 172, 183—185.

A COMPARISON of the influence of light of different colours on the third and sixth generations of *Azotobacter* as regards their nitrogen-fixing power. (Cf. J.C.S., March.)—W. G.

*Basic slag; Improvement of low-grade —.* W. S. Jones. Trans. Faraday Soc., 1920, 16, 324—327.

BASIC slag may be improved in quality by washing on a concentrating table and subsequently drying; in this way the phosphate content may be increased about 12%. By suitable tapping of the furnace, the slag may be separated into a richer and a poorer slag. Direct enrichment may be obtained by adding rock phosphate to the slag either in its molten condition or when cold before grinding. Frequently a low-grade slag is returned to the blast furnace with the next charge and its phosphorus taken up by the pig iron, from which it is obtained again in the Bessemer converter in the form of an enriched slag. The suggestion is made that the principle of the Bertrand-Thiel process might be utilised. In this process two furnaces are used, the pig iron being melted in the first without scrap before being run into the second containing the scrap etc. As no scrap is used in the primary furnace, the slag is not diluted thereby, and a grade containing a high percentage of phosphorus can be obtained.—J. H. J.

*Basic slags; Solubility of —. Part I. The reason why fluorspar in basic slag makes it relatively insoluble.* F. Bainbridge. *Part II. Solubility of basic slag in citric acid and carbon dioxide solvent.* J. E. Stead and E. W. Jackson. *Trans. Faraday Soc.*, 1920, 16, 302—309.

I. INCREASING amounts of fluorspar were added to a soluble slag and the solution curve determined. The solubility of the slag decreased rapidly and at a ratio of 8 calcium fluoride to 100 tricalcium phosphate became constant. A similar effect was produced in mixtures of calcium phosphate and silicate. In this case it is probable that a new compound consisting of lime, phosphoric acid, and calcium fluoride is formed. An open-hearth furnace slag was made with the addition of calcium fluoride and allowed to cool slowly. The interior of the mass consisted of needle-shaped crystals which upon analysis and crystallographic examination were found to be artificial apatite with some excess of fluoride and silicate. A solubility test on 5 g. of the crystals showed that 5% of the phosphate was soluble, and on 1 g. 26% (*cf. infra*). II. Four samples, namely, a soluble slag, an insoluble slag containing much fluorspar, a partly soluble slag containing little fluorspar, and artificial apatite crystals, were compared as to the solubility of their phosphate by the ordinary citric acid test and by repeated extraction with a saturated solution of carbon dioxide. It was found that, except in the partly soluble slag, long continued attack by carbonic acid dissolved out as much or more phosphoric acid than a single attack by citric acid, and that the solubility in either solvent was of the same order.—J. H. J.

*Basic slags; Comparison of the effect of various types of open-hearth — on grassland.* G. S. Robertson. *Trans. Faraday Soc.*, 1920, 16, 291—301.

FIELD experiments were made on three different soils under grass which were deficient in phosphate. Three varieties of basic slag were used: a high-grade Bessemer slag with 92% of soluble phosphate (on total phosphate), low-grade basic slags with 80—93% of soluble phosphate, and open-hearth basic slags with 20—45% of soluble phosphate. The hay crop was found to be as heavy on land treated with an open-hearth slag of 45% soluble phosphate as on land treated with the high-grade slags, but open-hearth slags with soluble phosphate below this amount gave much lighter crops. Clover crops were much increased by the application of open-hearth slag of 20% soluble phosphate, though the results were inferior to those with 90% soluble slags. A test of the citric solubility of the slags was made, using 1 g. of the sample instead of the usual 5 g. As a result, the citric solubility of the open-hearth slags was increased to over 60%, whilst the other basic slags showed 90—100% solubility. A Gafsa rock phosphate showed an increase from 38 to 89% solubility under the same conditions. In order to enable open-hearth basic slags of 5—8% phosphate content to be utilised as fertilisers it is suggested that they should be enriched during the process of manufacture by the addition of rock phosphate to the molten slag in the ladle.—J. H. J.

*Chloropicrin; Action of — on the germination of seeds.* E. Miège. *Comptes rend.*, 1921, 172, 170—173.

CHLOROPICRIN at a concentration of 20 c.c. per cb. m. of space is capable of destroying parasitic insects on seeds such as peas, beans, and wheat after 24 hrs. contact. Its action on the germination of the seeds varies with the species of seed. At this concentration it has very little effect on the germination of leguminous seeds or linseed, but in other cases it markedly reduces the germinative capacity and rate of germination of cereal, beet-

root, and hemp seeds. This effect increases with the concentration of the chloropicrin and the duration of contact, and under the conditions requisite for disinfection it reduces the germinative capacity of wheat by 30%.—W. G.

*Seed wheat; Injury to — resulting from drying after disinfection with formaldehyde.* A. M. Hurd. *J. Agric. Res.*, 1920, 20, 209—244.

SEED wheat which has been disinfected by treatment with 0.1% formaldehyde solution and kept in dry storage suffers considerable injury, but this injury can be prevented by sowing the seeds immediately after treatment. Experiments have now shown that the injury can be entirely prevented if the seeds are kept damp or are washed before being dried. As formaldehyde can be detected in seeds which have been dried, it is evident that the aldehyde remains on the seed as paraformaldehyde which gives off formaldehyde gas slowly in intimate contact with the seed. If the seed is stored in presence of an atmosphere of over 70% humidity, it is not injured, and in an absolutely dry atmosphere also there is no injury, but in intermediate humidities injury occurs. Formaldehyde solutions evaporated in moist atmospheres give no paraformaldehyde when the humidity is over 70%, but paraformaldehyde is found in increasing amounts in humidities below 60%. Untreated wheat is entirely killed in presence of strong formaldehyde solution in atmospheres of humidities over 30%. Apparently formaldehyde does not enter the seed as a solid nor as a gas, but in the state of solution. For seed injury to take place during dry storage sufficient moisture must be present to permit of paraformaldehyde being formed and of formaldehyde solution entering the seed. Barley is less susceptible than wheat, and the sorghums are quite uninjured even if 0.2% solution is used.—J. H. J.

*Sulphur dioxide; Prevention of damage to vegetation by absorbing — by charcoal.* Technischer Büro für die Chem. Ind. Chemotechnik, Wiesbaden. *Chem.-Zeit.*, 1921, 45, 96—97.

THE use of wood charcoal for the removal of sulphur dioxide from waste gases is recommended. Whereas water only absorbs 13.5% of its volume of sulphur dioxide from an atmosphere consisting entirely of the gas, and much less from a mixed atmosphere, charcoal absorbs 165 times its volume of the gas even from an atmosphere containing only small quantities of it. The recovery of the gas from the charcoal is also easy.—J. H. J.

## XVII.—SUGARS; STARCHES; GUMS.

*Beet slices; Dry distillation of exhausted —.* W. Paar and A. Starke. *Z. Ver. deuts. Zuckerind.*, 1920, 445—449.

ON dry distilling 200 g. of exhausted beet slices, 29—32 l. of gas (corresponding to 150 cb. m. per 1000 kg.) was obtained. This gas, which contained 16.0% CO, 20.9% CH<sub>4</sub>, and 15.4% H<sub>2</sub>, burnt with a luminous flame, and formed an explosive mixture with air and oxygen. It is suggested that the formation of gas of similar composition may explain the frequent explosions occurring in beet slice drying plants, local superheating of the product under the conditions obtaining not being unlikely.—J. P. O.

*Beet slices; Occurrence of saponins in exhausted —.* A. Traegel. *Z. Ver. deuts. Zuckerind.*, 1920, 449—459.

EXHAUSTED beet slices after drying were extracted with methyl alcohol, when the presence of neutral and acid saponins was established in the residue

obtained after removing the solvent, which residue was also proved to possess a strong hemolytic effect. The presence of saponins explains the death of fish in streams into which slice press waste waters have been discharged.—J. P. O.

*Beet sugar after-products; Crystallisation in the working of —.* A. Grill. Z. Ver. deuts. Zuckerind., 1920, 459–468.

SINCE the supersaturation of a solution of sucrose increases as the temperature falls, it is often necessary to add a certain quantity of water to the massecuite in the crystallisers while cooling to a suitable temperature for centrifuging, this quantity depending upon the temperature, the purity, and the density of the product concerned. The following table shows the Brix degree which the mother-syrup of a massecuite should possess for crystallisation without addition of water when centrifuging at temperatures between 35° and 60° C.

Apparent purity of syrup.	Temperature of centrifuging.				
	35°	42°	45°	50°	60°
	Bx.	Bx.	Bx.	Bx.	Bx.
57	85.4	87.2	87.5	88.2	90.7
58	85.7	87.5	87.7	88.5	90.9
59	86.0	87.7	88.0	88.7	91.1
60	86.3	88.0	88.3	89.0	91.3
61	86.6	88.3	88.5	89.2	91.5
62	86.9	88.5	88.8	89.5	91.7
63	87.2	88.8	89.0	89.7	91.9
64	87.5	89.1	89.3	90.0	92.1
65	87.8	89.3	89.6	90.2	92.3
66	88.1	89.6	89.8	90.5	92.5
67	88.4	90.0	90.1	90.7	92.7
68	88.7	90.2	90.4	91.0	92.9
69	89.0	90.4	90.7	91.2	93.1
70	89.3	90.7	90.9	91.5	93.3
71	89.7	91.0	91.2	91.8	93.5
72	90.0	91.3	91.5	92.0	93.7
73	90.3	91.6	91.8	92.3	93.9
74	90.6	91.9	92.0	92.5	94.1

When the syrup is so concentrated as to be without the range of values given in the table, the volume to which it must be diluted to bring its density to a suitable value is calculated, and the appropriate amount of water mixed with the contents of the crystallisers, preferably in three or four portions at a time, while continually stirring.—J. P. O.

*Vegetable decolorising carbons; Factors governing the decolorising effect of —.* A. B. Bradley. Int. Sugar J., 1921, 23, 25–32.

EXAMINATION of the effect of treating 50% solutions of raw Jamaica sugar with various decolorising carbons of different origin (adding 5% of the weight of syrup) showed that the greatest decolorising effect concurs with the most rapid rate of filtration. Moreover, in the case of 4 of the 5 samples tested (which included "Norit" and "Eponit") it was found that both the decolorising effect and the rate of filtration decreased as the amount of "dust" (that is, carbon passing a 124-mesh sieve) increased. This is true also in factory practice, filtration with fine or disintegrated carbon being much slower than with more uniformly coarse material, probably owing to the colloidal impurities forming a semi-pervious layer with the finer grains. Carbons having the greatest volume per unit weight give the best result in respect both of decolorising effect and rate of filtration; but equal volumes of different samples of decolorising carbons having the same degree of fineness do not give similar decolorising effects, the efficiency being probably influenced by the porosity of the material. When samples of decolorising carbons were treated successively with four portions of raw sugar liquor (without reactivation at each stage), the samples containing the

highest amount of "dust" were the most readily exhausted, and those which contained the maximum quantity of medium grain (that is, between 84- and 106-mesh) maintained their efficiency best.—J. P. O.

*Sugar mixtures containing two monosaccharides; Analysis of —.* C. A. Browne. Int. Sugar J., 1921, 23, 35–38.

VAN DER HAAR ("Anleitung zum Nachweis, zur Trennung, und Bestimmung der Monosaccharide," p. 133) has criticised the general principle of the author's method for analysing mixtures containing two reducing sugars (J., 1906, 446) on the ground that the reduction ratios of the sugars are not constant. It is, however, pointed out that in determining the reduction ratios Van der Haar averaged the extreme values indicating the reducing power of the various sugars, whereas the ratios taken at frequent regular intervals should have been averaged. On re-calculating the reduction ratios obtained by Van der Haar when using Schoorl's method, the figures found were in good agreement with those originally stated by the author (*loc. cit.*) when applying Allihn's method, and also with those found by Kjeldahl's procedure. It is concluded that the employment of reduction ratios in the analysis of sugar mixtures containing two monosaccharides is reliable for any of the ordinary methods of copper reduction with Fehling's solution; and that the ratios established for one method of reduction are applicable without serious error to other methods of reduction.—J. P. O.

*Sucrose; Experiments on the inversion constant of pure — in following the Clerget-Herzfeld double polarisation procedure.* O. Schrefeld. Z. Ver. deuts. Zuckerind., 1920, 402–408.

EXPERIMENTS carried out in 1910 showed that the factor for the calculation of the % sucrose in the Herzfeld modification of the double polarisation method should be about 133.0 at 20° C. when the temperature of hydrolysis is 60° C., the concentration 15 g. in 100 c.c., and the duration of heating 10–15 mins. (*cf.* Jackson and Gillis, J., 1920, 634 A.) —J. P. O.

*Invert sugar in a partially inverted and concentrated solution of sucrose; Tables for the approximate calculation of the —.* W. Paar. Z. Ver. deuts. Zuckerind., 1920, 409–413.

GIVEN the quotient of purity,  $Q$ , of the syrup before inversion, the Brix degree,  $B$ , after inversion and concentration, and the polarisation,  $P$ , of the syrup also after inversion and concentration, the invert sugar per cent.  $I = 0.00754(B \times Q - 100P)$ ; or where the total sugar per cent.,  $T$ , is known,  $I = (T - P) 0.754$ . Based upon these equations, tables have been constructed for the ready calculation of the invert sugar per cent. in the case of syrup of 90° and 95° quotient of purity, the Brix degree varying from 50° to 80°, and the polarisation from +70° to -20°.—J. P. O.

*Calcium salts in sugar juices and syrups; Determination of — volumetrically, using standard soap solution.* W. Montgomery. Int. Sugar J., 1921, 23, 39–42.

TABLES are given for the calculation of the calcium oxide as % of total solids (Brix), given the c.c. of soap solution (1 c.c. of which = 0.001 g. CaO), and the Brix of the juice or syrup examined.—J. P. O.

#### PATENT.

*Sugar factories; Manufacture of a precipitate [feeding stuff] rich in albumen and poor in ash from the raw juices and waste liquors of [beet] —.* M. von Wierusz-Kowalski. E.P. 132,798, 16.9.19. Conv., 11.7.16.

THE liquid, heated to 80°–100° C., is treated with



a very small amount (0.01–0.02%) of sulphurous acid, and then, at about 80° C., with sufficient lime or other base to produce an alkalinity of 0.02–0.04%. The precipitate obtained contains about 20% of protein, 5% of phosphoric acid, and 10% or less of ash, and may be employed as a feeding stuff.—J. H. L.

### XVIII.—FERMENTATION INDUSTRIES.

*Malt; Simplification of the Lintner-Wirth method for determining the diastatic power of —.* G. Roeder. *Woch. Brau.*, 1921, 38, 5–7.

THE procedure suggested comprises the same operations as in the Institute of Brewing Committee method (J., 1906, 236), but the malt extract is allowed to act on the starch solution for  $\frac{1}{2}$  hr. at 17.5° C. instead of 1 hr. at 70° F. (21° C.), the reducing sugar is determined by Kjeldahl's gravimetric method, and the diastatic power is expressed as g. of maltose per 100 g. of malt.

—J. H. L.

*Malts; Determination of the extract of coloured —.* C. G. Matthews. *J. Inst. Brew.*, 1921, 27, 22–25.

THE Institute of Brewing Committee method (J., 1910, 1323) has several practical disadvantages, and usually gives somewhat low results. The following has been found more convenient and accurate:—A mixture of 40 g. of standard malt (D.P. 35°–45° Lintner), ground to 25° Seck, and 10 g. of the sample of crystal malt, similarly ground, is mashed by the Committee method, and a bright filtered wort obtained in the usual way. Then (sp. gr. of wort–4/5 sp. gr. of 10% wort from the standard malt)  $\times 5$  = sp. gr. due to sample; and this multiplied by 3.36 gives the extract yield of the sample. Further, (colour of the wort in a 1 in. coll–4/5 colour of 10% wort from the standard malt)  $\times 5$  = colour of sample in 10% wort; this multiplied by 10 gives the total colour units referred to the sample itself. The method is applicable to all coloured malts and also to flaked materials. Some comparative results with this and the Committee method are given in tables.—J. H. L.

*Maize and rice and the method of using them [in brewing].* W. Windisch. *Woch. Brau.*, 1921, 38, 9–15.

IN view of the fact that German brewers are now permitted to employ maize grits, free from husks and embryos, and also rice products unsuitable for human consumption, the author gives an account of the composition, method of production, and brewing properties of these adjuncts, and also discusses the manner in which they may be employed in brewing. Some experiments on the malting of maize are also described.—J. H. L.

*Cellulose; Action of dilute mineral acids on —.* A. Wohl and K. Blumrich. *Z. angew. Chem.*, 1921, 34, 17–18.

THE insoluble residue (hydrocellulose) remaining after prolonged heating of cellulose with dilute acids is of the nature of a reversion product rather than an intermediate degradation product; such residues are often more resistant to hydrolysis than the original cellulose, and unsuitable for fodder owing to their indigestibility. In the hydrolysis of cotton cellulose by 0.5–3% hydrochloric acid at 100° C., the cupric-reducing powers of the residue and the solution were determined at different stages. When, after a certain period of heating, the solution was separated from the residue and both were further heated separately with acid of the same concentration, more total reducing sugar

was formed than when the heating was continued without any such separation. It is concluded that reducing sugars which have already passed into solution interact, on continued heating, with the residue in the colloidal phase, and form reversion products.—J. H. L.

*Cellulose; Saccharification of —.* A. Wohl and H. Krull. *Cellulosechemie*, 1921, 2, 1–7.

IN laboratory experiments cellulose (cotton) yielded 97% of the theoretical amount of reducing sugar by a preliminary liquefaction (primary hydrolysis) with fuming hydrochloric acid, followed by saccharification (secondary hydrolysis) by boiling with dilute acid. The best method of procedure was as follows: The material was moistened with 3 pts. of water and then cooled by means of ice and saturated with hydrogen chloride. It was then left for 5 hrs., or longer, at 20° C., after which nearly the whole of the acid was removed by evaporation *in vacuo* at temperatures up to 70° C. The residue was dissolved in water and the solution, containing about 10% of material and 1% of hydrochloric acid, was boiled for 8 hrs. The sugar was determined as dextrose by cupric reduction. Results obtained with a variety of products indicate that the method might be employed for the determination of cellulose. 100 g. of pine shavings (dry substance) gave 60.9 g. of apparent dextrose from which 18 g. of alcohol was produced on fermentation. This yield of alcohol is only about 60% of the theoretical, whilst the reducing sugars obtained from pure cellulose gave alcohol equivalent to 90% of the theoretical. Probably the hydrolysis of the cellulose in wood is impeded by incrusting substances, and, on the other hand, if the duration of the primary hydrolysis is prolonged to 24 hrs. non-fermentable reversion products are formed in relatively large quantities (cf. preceding abstract). All attempts to improve the process by a preliminary treatment to dissolve the lignin were unsuccessful; the yield of reducing sugars was increased, especially by chlorine treatment, but the yield of alcohol was diminished. An 18% yield of alcohol from pine wood, however, would entitle the process to serious consideration if only the difficulties associated with the evaporation of the hydrochloric acid on a large scale could be overcome.—J. H. L.

*Wine; Determination of sugar in —.* W. Fresenius and L. Grünhut. *Z. anal. Chem.*, 1920, 59, 415–457.

A SCHEME of analysis is proposed, to replace the present German official methods. The main features are as follows:—Samples used for the determination of sugars are de-alcoholised by evaporation to one-third their volume, after neutralisation. The reducing and rotatory powers are in general determined after clarification with basic lead acetate and removal of excess of lead by sodium sulphate; but for dry wines animal charcoal may be employed, to avoid dilution. For the hydrolysis of sucrose the Clerget-Herzfeld procedure is adopted, and before the subsequent clarification with basic lead acetate the hydrochloric acid is precipitated by a slight excess of silver nitrate; an aliquot part of the filtrate from the silver chloride is almost neutralised with sodium hydroxide and then treated with basic lead acetate, the excess of lead and silver being afterwards removed by means of sodium phosphate. Reducing sugars are determined by the gravimetric method, except in dry wines containing less than 5 g. per litre, for which Schoorl's volumetric method (J., 1899, 791) is used. Formulae based on Woy's equations (*Z. öfentl. Chem.*, 1898, 4, 33) are given for calculating the amounts of dextrose and levulose present in wine from the reducing and rotatory powers as determined by the methods described.

—J. H. L.



**Methyl alcohol; Detection of — in spirits.** F. Rabe. Pharm. Zeit., 1921, 66, 72.

THE formaldehyde resulting from the oxidation of methyl alcohol may be identified by means of morphine-sulphuric acid reagent; in preparing the latter by mixing morphine hydrochloride with sulphuric acid care should be taken to expel all hydrochloric acid by heating the mixture, otherwise a red coloration develops in the reagent itself and renders it useless. Resorcinol-sulphuric acid reagent (0.2 g. per 10 c.c.) is recommended for the detection of formaldehyde.—W. P. S.

**Action of dyes on yeast.** Fraser. See XIXB.

#### PATENTS.

**Beer or other fermented liquors; Process of brewing or preserving —.** R. de Fazi. E.P. 143,506, 12.5.20. Conv., 9.10.19.

BEER is treated, after the fermentation, with about 0.01–0.02% of hexamethylenetetramine or derivatives thereof.—J. H. L.

**Alcohol; Recovering — from fermented liquids and apparatus therefor.** W. G. Toplis. U.S.P. 1,364,160, 4.1.21. Appl., 27.7.17.

THE waste liquid (distillation residue) from a primary distillation apparatus is subjected to a secondary distillation, and the alcohol obtained from the latter is mixed with the supply of original liquid before this liquid enters the primary distillation apparatus.—J. H. L.

**Sulphite-cellulose waste lyes.** U.S.P. 1,342,721. See V.

### XIXA.—FOODS.

**Lard; Detection of adulteration of — with fats containing tristearin.** R. H. Kerr. J. Assoc. Off. Agric. Chem., 1920, 4, 195–201.

FIVE g. of the lard is diluted to 25 c.c. with warm acetone in a stoppered cylinder, and the mixture is kept at 30° C. for 18 hrs.; the liquid is then decanted and the mass of crystals at the bottom of the cylinder is washed twice with warm acetone, using 5 c.c. each time and taking care not to break up the deposit. The latter is shaken with a further 5 c.c. of warm acetone, transferred rapidly to a small filter, again washed with acetone, and the excess of the latter removed as far as possible by suction. The crystalline deposit is spread on paper, dried, and the m.p. determined. A m.p. below 63.0° C. indicates adulteration. The fatty acids are then obtained from the crystals in the usual way and their m.p. determined. If the m.p. of the glycerides (crystalline deposit) plus twice the difference between the m.p. of the glycerides and the m.p. of the fatty acids is less than 73° C., the lard is adulterated.—W. P. S.

**Histidine [in proteins]; New method for the determination of —.** W. E. Thrun and P. F. Trowbridge. J. Assoc. Off. Agric. Chem., 1920, 4, 194–195.

A AROMINE absorption method is described for use in conjunction with the Van Slyke method (J., 1911, 1135). An aliquot portion of the solution containing the bases of a coagulable protein is treated with bromide-bromate solution and hydrochloric acid, and, after 15 mins., the excess of bromine is titrated with thiosulphate solution. Sulphur is determined in another portion of the solution and the quantity of cystine present is calculated from the sulphur content. One mol. of cystine absorbs 10 atoms of bromine, whilst 1 mol. of histidine absorbs somewhat more than 2 atoms of bromine; consequently

a factor for the bromine absorption of histidine is necessary.—W. P. S.

**Ammonium citrate; Behaviour of neutral — in certain phosphate solutions. [Determination of lead in phosphate baking powders.]** H. E. Patten and G. H. Mains. J. Assoc. Off. Agric. Chem., 1920, 4, 235–237.

IN the determination of lead in phosphate baking powders the addition of neutral ammonium citrate to the hydrochloric acid solution of the powder prevents the precipitation of calcium phosphate when the solution is treated with ammonia. At *p* values below 5.0 calcium citrate remains in solution; between 5.0 and 7.0 the precipitation of calcium citrate is very slow, but at 7.1 an immediate permanent precipitate of gelatinous calcium citrate is formed.—W. P. S.

#### PATENTS.

**Foods from kitchen waste and the like; Manufacture of animal —.** R. L. D. Taylor. E.P. 140,812, 26.3.20. Conv., 25.2.19.

KITCHEN waste is sorted, pressed to remove excess water, and mixed with a vegetable filler, such as oat hulls. The mixture is passed through a pulping machine and then down an inclined rotary dryer heated by steam at 352° F. (178° C.), which converts the pulp into nodules. From the dryer the material falls on to a belt conveyor passing through an oven at 400° F. (about 200° C.), which completely cooks and sterilises it. On leaving the oven the material passes through a screening device to separate the nodules of  $\frac{1}{4}$ – $\frac{1}{2}$  in. diameter, which are suitable to be used as a scratch food for poultry. The coarser and finer nodules are collected separately and ground, and serve as a meal for making into dog biscuit.—J. H. J.

**Margarine; Manufacture of —.** J. E. Green, jun. E.P. 156,000, 9.1.20.

HARD fat is placed in a closed jacketed vessel supported on trunnions, and steam is passed through the jacket to melt the fat, which is stirred continually by a gauze mixer in order to collect fibrous material. The mixer is afterwards removed with the adhering fibrous material, and is cleaned and replaced. Cold soured milk and oil are introduced into the vessel and the mixer rapidly rotated until a uniform product is formed. This is cooled by passing a cooling medium through the jacket, and agitation is continued until the whole thickens to a homogeneous emulsion to which salt and preservatives are added, and agitation completed at a high speed.—J. H. J.

**Bread-making; Composition of materials adapted for use in —. Method of manufacturing dough ingredients. Method of producing materials for bread-making and composition of materials for use as a bread-dough ingredient.** R. L. Corby, Assr. to The Fleischmann Co. U.S.P. (A) 1,355,127, (B) 1,355,128, and (C) 1,355,129, 12.10.20. Appl., (A) 15.7.15, (B) and (C) 15.6.17. Renewed, (A) 5.2.20, (B) 29.7.20, (C) 30.7.20.

(A) A PRODUCT such as is obtained by the process described under (a). (B) A cereal mash is saccharified, and then acidified (with lactic acid) sufficiently not only to render the diastatic and proteolytic enzymes inactive but also to condition the gluten of the dough; after filtering from undissolved and coagulated matters, the filtrate is evaporated. (C) Invert sugar is mixed with the product before evaporation.—J. H. L.

**Butterin, oleo-margarine and the like; Means for making —.** N. D. Nielsen. U.S.P. 1,364,297, 4.1.21. Appl., 18.4.18.

AN emulsifying tank and a crystallisation vessel are

connected by a duct which enters the latter near the bottom and which is provided with a pump so that emulsion from the tank may be forced into the crystallisation vessel. A propeller within the vessel, near the inlet of the duct, enables the emulsion to be maintained in the lower part of the vessel, but permits crystals formed from the particles of the emulsion to rise to the surface. Means are also provided for forcing cold water into the vessel and for maintaining the contents of the latter at a temperature below 40° F. (4° C.).—J. H. L.

*Foods and other substances; Apparatus for heat-treating* —. N. H. Fooks. U.S.P. 1,363,103, 21.12.20. Appl., 22.12.19.

A RETORT is provided with inlet and outlet orifices for liquid, means for admitting gas under pressure at a point within the upper part, means for heating, and charging and discharging ports at the top. Within the retort is a rotary spiral conveyor by which objects are carried by a tortuous path from the charging to the discharging port. There are also air-locks at the ports and means for moving the objects through the air-locks.—J. H. L.

*Food preparation and process of making same.* F. E. Coombs, Assr. to J. F. Judge. U.S.P. 1,363,193, 21.12.20. Appl., 10.10.19.

A COOKED, desiccated starchy food of loose-grained character, the grains of which are superficially free from starch, is prepared by treating the surface of the grains of the cooked material with a diastatic solution, then destroying the diastase by heat, and finally drying and browning the product.—J. H. L.

*Chocolate; Process of making soluble* —. J. Friedman. U.S.P. 1,364,192, 4.1.21. Appl., 2.4.19.

A SOLUBLE preparation capable of being moulded and of withstanding a relatively high temperature without softening, is produced by boiling syrup, adding a liquid extract, cooling, powdering the solidified mass, and mixing it with powdered cacao beans and fat.—J. H. L.

*Casein precipitate [desiccated milk food]; Desiccated* —. P. W. Turney. U.S.P. 1,364,417, 4.1.21. Appl., 19 4.18.

MILK is curdled by an enzyme to such a degree that a flocculent precipitate is produced, the activity of the enzyme is then arrested by cooling, the product is sprayed into a chamber maintained at a low temperature and under partial vacuum, and dehydrated by means of air currents.

*Desiccating [milk]; Process and apparatus for* —. I. S. Merrell, Assr. to Merrell-Soule Co. U.S.P. 1,365,055, 11.1.21. Appl., 30.10.16.

MILK is caused to flow spirally down the interior surface of a heated cylinder, and an elevating force is exerted upon the liquid to retard its flow.

—L. A. C.

*Rice; Process for treating* —, and product thereof. M. M. Baumgartner. U.S.P. 1,364,912, 11.1.21. Appl., 11.8.19.

HULLED rice grains are steamed under such conditions as to sterilise them and dextrinise their starchy contents without cracking them; they are then dried so as to resemble untreated rice in form, bulk, and moisture-content.—J. H. L.

*Coffee; Method of making soluble* —. H. Anhaltzer. U.S.P. 1,365,443, 11.1.21. Appl., 9.11.18.

A SOLID coffee preparation is produced by steaming roasted ground coffee beans, then extracting with alcohol, and finally distilling the extract. All these operations are carried out with exclusion of air, in order to prevent loss of volatile oils.—J. H. L.

*Food enclosed in sealed cans and other containers; Process of heat-treating bodies, such as* —. N. H. Fooks. E.P. 156,994, 22.1.20.

SEE U.S.P. 1,331,337 of 1920; J., 1920, 311 A.

*Tea-drying apparatus.* H. A. Tempelaar. U.S.P. 1,364,463, 4.1.21. Appl., 23.8.20.

SEE E.P. 151,461 of 1919; J., 1920, 760 A.

*Foodstuff; Animal* —. F. W. Gee. U.S.P. 1,365,393, 11.1.21. Appl., 13.11.19.

SEE E.P. 136,237 of 1918; J., 1920, 170 A.

*Feeding stuff.* E.P. 132,798. See XVII.

## XIXB.—WATER PURIFICATION; SANITATION.

*Waters; Determination of hydrogen sulphide in natural* —. Sensitiveness of the starch iodine reaction. E. Chrétien and H. Vandenbergh. Ann. Chim. Analyt., 1921, 3, 19—23.

ONE litre of the water is treated with 20 c.c. of saturated barium chloride solution, filtered, and 510 c.c. of the filtrate is added to a mixture of 10 c.c. of N/100 iodine solution and 0.1 g. of potassium iodide; 10 c.c. of N/100 thiosulphate solution is then added and the excess of this is titrated with N/100 iodine solution. With 500 c.c. of distilled water free from ammonia and nitrous acid, 0.05 g. of potassium iodide free from iodate, and 1 c.c. of 1% starch paste, a distinct blue colour is produced with 0.10 c.c. of N/100 iodine at 10° C., whereas at 19° and 28° C., respectively 0.15 and 0.25 c.c. are required and the coloration is less distinct.

—W. P. S.

*Mercuric chloride; Toxicity of* — and its solubility in aqueous alcohol. J. S. Laird. J. Phys. Chem., 1920, 24, 736—737.

THE solubility of mercuric chloride in aqueous alcohol shows a pronounced minimum at a concentration of 24% of alcohol and this corresponds closely to the maximum toxicity of such solutions as observed by Paul and Krönig (Z. physik. Chem., 1896, 21, 448).—W. G.

*Phenol solutions containing sodium benzoate; Toxicity towards Staphylococcus of dilute* —. K. E. Burgess. J. Phys. Chem., 1920, 24, 738—740.

DETERMINATIONS of the rate at which staphylococci are poisoned by dilute solutions of phenol with and without the addition of sodium benzoate confirm the conclusion that the abnormalities observed by Lemon (cf. J., 1920, 799 A) were due to plasmolysis.

—W. G.

*Yeast; Action of Methylene Blue and certain other dyes on living and dead* —. C. G. Fraser. J. Phys. Chem., 1920, 24, 741—748.

THE behaviour of nine different dyes, namely, Grüber's Methylene Blue, Fuchsin, Congo Red, Erythrosin, water-soluble Safranin, Merck's Methylene Blue, Gentian Violet, Methyl Green, and Kahlbaum's Methylene Blue 6 B extra, with living yeast and with yeast killed either by boiling or by the action of phenol, was examined in order to find a convenient criterion of death in quantitative toxicological investigations with microbes. Erythrosin and Methylene Blue 6 B extra gave the best results. In a solution of extract of malt to which Methyl Green has been added, yeast cells may lose their power of reproduction without becoming stained.—W. G.

*Bacteria; Method for investigating the action of ultra-violet rays on* — T. Mashimo. Mem. Coll. Sci. Kyoto, 1919, 4, 1—11.

A THIN layer of agar culture medium spread over a glass plate and uniformly inoculated with the bacteria under investigation, was exposed to the ultra-violet spectrum in a quartz spectrograph, and afterwards incubated. The parts sterilised by the rays became easily distinguishable from the rest of the plate by the absence of bacterial growth, and the spectral image thus obtained was fixed by exposure to formaldehyde vapour. For all the well-known species of non-pathogenic bacteria investigated, the bactericidal portion of the spectrum was the same, viz., from 295  $\mu$  to 186  $\mu$ , the region of greatest activity being about 275  $\mu$ .—J. H. L.

*Basic exchange.* Ramann and Junk. See VII.

*Chloropicrin and seeds.* Miège. See XVI.

*Disinfecting seed wheat.* Hurd. See XVI.

*Absorbing sulphur dioxide.* See XVI.

#### PATENTS.

*Filtering apparatus [for water].* W. Paterson. E.P. 156,270, 2.3.18.

IN order that each filter in a battery of filters may receive water in proper proportion to its filtering area, it is provided at one side of the space above the filtering medium with a pocket into which the incoming water or precipitation tank effluent falls, passing over a weir as it does so, and from which it flows away into the filter through an opening in the bottom. The overflow from the bottom of the filter is controlled by a valve floating in the liquid over the filter medium. As the level of the water over the medium rises, the float valve opens the outlet until the outflow and the inflow are the same. This continues until the filter begins to become choked, when the valve opens further. Thus the water level gradually rises over the medium until it is flush with the weir in the pocket. In order to prevent splashing in the pocket, the opening between the pocket and the filter may be controlled by a float valve in the pocket which keeps the level of the water just below the weir.—J. H. J.

*Sand-filters.* N. McK. Barron. E.P. 156,424, 1.10.19.

IN a battery of sand filters, the filters are arranged circumferentially around a sand pit, and clogged sand from the filters is passed by means of a pressure ejector into the pit, from which it is lifted and washed in a combined operation by means of a counterflow of water. The washed sand is delivered into a container situated at a level above the top of the filters, and is distributed to the filters as required. The sand container is of such a size as to hold sufficient sand to fill one of the filters and to leave a reserve supply in addition. The container has a tubular extension in its lower portion, the sand in which remains undisturbed when clean sand is delivered into the container; the pipe connecting the container with a filter proceeds from this extension.—J. H. J.

*Ozone water-purifying apparatus.* H. B. Hartman, Assr. to Electric Water Sterilizer and Ozone Co. U.S.P. 1,363,589, 28.12.20. Appl., 24.1.20.

AN apparatus for purifying water by means of ozone includes a storage reservoir, an ozone generator, and automatic means for synchronously controlling the flow of raw water and ozone to the reservoir. —J. H. J.

*Sewage; Method of and apparatus for treating* — C. P. Landreth. U.S.P. 1,364,387, 4.1.21. Appl., 11.1.17.

SEWAGE is aerated and a precipitant is added; the precipitate is allowed to settle, and the sludge is withdrawn, mixed with suitable finely divided material, and de-watered.—J. H. J.

*Sewage; Method of treating* — J. P. Ball. U.S.P. 1,364,676, 4.1.21. Appl., 22.10.18.

SEWAGE sludge is subjected to a forced current of air to remove noxious gases, which are passed through a collecting medium to remove the entrained particles of liquid.—J. H. J.

*Germicide.* C. A. Weeks; L. H. Weeks extrix. U.S.P. 1,366,106, 18.1.21. Appl., 15.6.18.

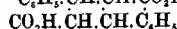
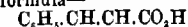
SEE E.P. 134,880 of 1918; J., 1920, 80 A.

*Composition for liberating sulphur dioxide.* U.S.P. 1,356,029—30. See VII.

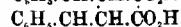
### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Truxillic and truxinic acids; Natural and synthetic* — R. Stoermer and E. Laage. Ber., 1921, 54, 77—85.

THE isolation of a seventh isomeride of truxillic acid, neotruxinic acid, induces the authors, for the sake of simplicity, to designate only those acids derived from the formula—



as truxillic acids, whilst those derived from the formula—



are termed truxinic acids. The truxinic acids can be conveniently separated from the truxillic acids by taking advantage of the fact that the potassium and ammonium salts of the former are very sparingly soluble in cold alcohol, in which the truxillic acids dissolve freely. The natural mixture of truxillic acids obtained during the isolation of cocaine yields under the most favourable conditions  $\alpha$ -truxillic acid (25%),  $\epsilon$ -truxillic acid (4.25%),  $\beta$ -truxinic acid (29%),  $\delta$ -truxinic acid (10%), neotruxinic acid (0.12%); the remainder consists of benzoic and cinnamic acids mixed with considerable quantities of resinous acids. The presence of  $\gamma$ -truxillic or  $\zeta$ -truxinic acid could not be established. (Cf. J.C.S. March.)—H. W.

*Fumaric acid and inositol; Occurrence of* — L. Zechmeister and P. Szécsi. Ber., 1921, 54, 171—173.

FUMARIC acid in the form of the acid potassium salt,  $2KC_4H_3O_4.H_2C_6O_5$ , has been isolated from the alcoholic extract of *Capsella bursa pastoris*.  $r$ -Inositol, m.p. 218°—219° C., has been obtained from the aqueous extract; it is present to the extent of at least 0.03% of the dried drug.—H. W.

*Hydrogenation of aromatic compounds by the help of platinum.* IV. Dependence of catalytic hydrogenation on the presence of oxygen. R. Willstätter and E. Waldschmidt-Leitz. Ber., 1921, 54, 113—138.

CATALYTIC hydrogenation cannot be effected by spongy or colloidal platinum or palladium or by nickel in the complete absence of oxygen; the metals can be activated by agitating their suspensions with air. The active catalysts contain both hydrogen and oxygen, the latter of which is gradually removed during the course of hydrogenation under normal conditions, and rapidly by certain contact

poisons such as glycerol and thiophen, although it remains doubtful if all cases of poisoning are due to this cause. The technique of catalytic hydrogenation can be considerably improved since the catalyst, when showing signs of diminished activity, can be reactivated by treatment with oxygen, and poisoning may be circumvented either by the same procedure or by increasing the amount of catalyst; priming with oxygen is particularly useful when the metal is used repeatedly or in small quantity. The use of spongy palladium has no advantage over that of platinum, whilst, on the other hand, its greater absorptive capacity for hydrogen renders re-activation by oxygen more difficult to such an extent that the process becomes either dangerous or tedious. (Cf. J.C.S., Mar.)—H. W.

*Chloroacetic acids; Catalytic decomposition of* — J. B. Senderens. *Comptes rend.*, 1921, 172, 155—158.

WITH thorium oxide or kaolin as catalyst, monochloroacetic acid is decomposed at 220° C., giving hydrogen chloride, carbon monoxide and dioxide, and carbon. Trichloroacetic acid gives chloroform, tetrachloroethylene, hexachloroethane, hydrogen chloride, carbon monoxide and dioxide. With animal charcoal as catalyst monochloroacetic acid decomposes as above, but in the case of trichloroacetic acid almost the only change is the formation of chloroform and carbon dioxide. (Cf. J.C.S., March.)—W. G.

*Propenol [allyl alcohol]; Mode of pyrogenic decomposition, at high temperature, of* — E. Peytral. *Bull. Soc. Chim.*, 1921, 29, 39—44.

In the sudden decomposition of propenol at high temperatures the two primary reactions are  $\text{CH}_3\cdot\text{CH}\cdot\text{CH}_2\text{OH} = \text{CH}_2\cdot\text{CH}\cdot\text{CHO} + \text{H}_2$ , and  $\text{CH}_3\cdot\text{CH}\cdot\text{CH}_2\text{OH} = \text{CH}_3\cdot\text{C}\cdot\text{CH}_2 + \text{H}_2\text{O}$ . For every three molecules of propenol decomposed, two yield acrolein and one gives propylene. The acrolein undergoes a secondary change, giving ethylene and carbon monoxide.—W. G.

*Formic acid; Sudden pyrogenic decomposition of — and the preparation of carbon monoxide.* J. A. Muller and E. Peytral. *Bull. Soc. Chim.*, 1921, 29, 34—39.

WHEN formic acid is suddenly decomposed by passing it rapidly through a platinum tube at 1150° C., the primary action is the formation of carbon dioxide and hydrogen. The production of carbon monoxide and water is due to the reduction of the carbon dioxide by the hydrogen. At a sufficiently high pressure the velocity of this secondary reaction may become so great as to make it appear as if it were the primary reaction.—W. G.

*Eucalyptol; o-Cresol method for the determination of* — C. T. Bennett and M. S. Salamon. *Perf. Essent. Oil Rec.*, 1921, 12, 11—12. (Cf. J., 1920, 832 A.)

AFTER further experience the authors modify their criticism of the method (J., 1920, 704 A) and agree with Cocking that it affords a rapid and easy means of determining eucalyptol in all grades of eucalyptus oil provided that they contain not less than 45% of this constituent.—W. P. S.

*Acetic acid; Binary and ternary mixtures obtained in the synthetic manufacture of* — P. Pascal, Dupuy, Ero, and Garnier. *Bull. Soc. Chim.*, 1921, 29, 9—21.

THE two processes of manufacture considered are the direct oxidation of acetaldehyde mixed with acetic acid and the electrolytic oxidation of acetaldehyde or of paraldehyde in sulphuric acid solution. The densities and boiling-points of binary mixtures of water and acetic acid; water and acetaldehyde; and

acetaldehyde and acetic acid, and of ternary mixtures of acetaldehyde, acetic acid, and water are given, and alongside the boiling-points of the various mixtures the composition of the liquid and of the vapour is given in each case. The boiling-points of ternary mixtures of acetic acid, sulphuric acid, and water and the composition of the liquid and vapour in each case are also given.—W. G.

*Menthone; Electrolytic reduction of* — M. Matsui and S. Shimizu. *Mem. Coll. Sci., Kyoto*, 1920, 4, 245—264.

MENTHONE can be reduced electrolytically to menthol in acid solution (sulphuric or hydrochloric). The best results were obtained using an amalgamated tin or lead cathode, the cathode solution consisting of a mixture of 75% anhydrous acid (40—45 c.c.) with 94% alcohol (35—40 c.c.) at a temperature below 15° C. The current used was 6—8 ampères per 100 sq. cm. at 11—13 volts. The yield of menthol was only 25% of the theoretical and the product was difficult to purify. An oily by-product was always obtained, especially when more dilute acid was used. This substance is a more highly reduced product and may be menthane.—E. H. R.

*Lemon-seed oil.* Bertolo. See XII.

*Saponins in beet.* Traegel. See XVII.

#### PATENTS.

*Cymene; Manufacture of para-* — British and Foreign Chemical Producers, Ltd. From Rheinische Kampferfabr. G.m.b.H. E.P. 156,325 6.10.19. Addn. to 142,738 (J., 1920, 501 A).

THE terpenes of b.p. about 160° C. present in pine-needle oil are treated as described in the chief patent. The yield of p-cymene is increased if the terpenes are heated with an inorganic or organic acid or acid salt, and, after separation from the acid, purified by fractional distillation, using the fraction of b.p. 175° C. for subsequent treatment.—L. A. C.

*Reactive acid liquor from hydrocarbon gases containing olefines; Process of making* — (a) Acid liquor and process of producing same. (c) Process of treating gasoline and the like. (d) Separation process of making alcohols. (e) Process of purifying alcoholic material. (f) Process of separating alcohols and hydrocarbons. (g) Solvent. (h) Direct production of ester bodies from unsaturated hydrocarbons. (i) Ester bodies from unsaturated hydrocarbons. (j) Process of directly esterifying olefines and product thereof. (k) Process of making ketone bodies. (A) M. D. Mann, jun., and R. R. Williams, (e, p & q) C. Ellis, (o) M. D. Mann, jun., (x) M. J. and G. Cohen, A. Breslauer and H. Asher, (r, s, t, and v) C. Ellis and M. J. Cohen, (u) C. Ellis and A. A. Wells, Assrs. to S. B. Hunt. U.S.P. 1,365,043—53, 11.1.21. Appl. (A) 11.3.19, (B) 26.6.17, (C) 6.7.18, (D) 5.6.18, (E) 8.6.18, (F and T) 25.4.18, (O) 8.1.18, (X) 25.6.17, (J) 30.6.16, and (K) 18.3.18.

(A) THE gases are brought into intimate contact with sulphuric acid in the presence of an inactive absorbent for the gases. (S) Hydrocarbon material containing olefines and liquid saturated hydrocarbons is treated with sulphuric acid, and the product is used for treating more olefine material to obtain an acid liquor saturated with olefines. (C) Sulphuric acid is gradually brought into contact below 20° C. with an intimate mixture of unsaturated hydrocarbons and a large proportion of a diluent for the acid. (D) A mixture of alcohols and sulphuric acid is treated with a material which

dissolves the alcohols but not the acid. (g) Alcohols are purified by distillation in the presence of a small quantity of sulphuric acid. (h) A mixture of alcohols and hydrocarbons is treated with sulphuric acid of the correct strength to dissolve the alcohols. (i) A solvent boiling mainly below 110° C. is composed of a mixture of propyl monoacetate with small quantities of ethyl and butyl monoacetate. (j) The product obtained by treating an olefine with sulphuric acid is treated with a substance capable of replacing the sulphuric acid radicle with an organic acid radicle. (k) A liquid material consists mainly of a mixture of unsubstituted mono-esters of monohydric alcohols corresponding to the olefines of cracked petroleum. (l) A fatty acid is heated with an olefine in the presence of an acid catalyst. (m) Cracked gasoline containing unsaturated hydrocarbons is treated with sulphuric acid of sp. gr. 1.8, residual gasoline is separated by gravity, and the acid material is oxidised electrolytically to produce ketones.

—L. A. C.

*Mercury compounds of glucosides and process of making same.* M. Hartmann, Assr. to Soc. of Chem. Ind. in Basle. U.S.P. 1,354,105, 28.9.20. Appl., 29.4.20

By heating glucosides, such as amygdalin, arbutin, salicin, etc., with mercuric acetate solution, mercury derivatives are formed in which the mercury cannot be detected by means of soda lye. The products are white powders, readily soluble in water, but soluble only with great difficulty in organic solvents. They are only feebly toxic, do not produce cutaneous necroses, and are suitable for injections.

*Pinacone diacetate; Preparation of* —. Farbenfabr. vorm. F. Bayer und Co. G.P. 327,123, 14.11.16.

AN almost quantitative yield of pinacone diacetate is obtained by treating a pinacone with acetic anhydride in the presence of a sulphonic acid, such as anthraquinone-1.5-disulphonic acid, toluenesulphonic acid, or naphthalenesulphonic acids, or of other strong acids, such as sulphuric acid.

—A. J. H.

*Aluminium tannate; Preparation of basic* —. Chem. Fabr. auf Aktien (vorm. E. Schering). G.P. 328,341, 18.7.18.

A SOLUTION of alkali tannate is treated with an aluminium salt with addition of sufficient caustic alkali to keep the solution alkaline. The basic aluminium tannate thus prepared is a grey, odourless and tasteless powder, having the probable formula  $(C_{12}H_6O_6 \cdot Al(OH)_3)_n \cdot C_{12}H_6O_6 + 12H_2O$ , which is insoluble in cold water, soluble in acids and alkalis, and insoluble in hydrochloric acid having an acidity comparable to that of stomach juices.

—A. J. H.

*Glycerin substitute.* Akt.-Ges. f. Anilinfabr. G.P. 328,530, 15.11.18.

SOLUTIONS of salts are rendered much more viscous, and suitable for use as glycerin substitutes, by the addition of betaine. A mixture of 180 pts. of calcium chloride, 350 pts. of water, and 465 pts. of betaine has sp. gr. 1.2485 which is equal to that of 94.5% glycerin.—A. J. H.

*Benzoic acid; Production of* —. J. B. Senderens, Assr. to Poulenc Frères. U.S.P. 1,365,956, 18.1.21. Appl., 7.7.19.

SEE E.P. 143,392 of 1919; J., 1920, 527 A.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Photochemical processes; Influence of temperature on* —. J. Plotnikow. Z. wiss. Phot., 1921, 20, 125—139.

NON-ILLUMINATED reactions have temperature coefficients which increase with decreasing temperature and approach unity at very high temperatures. The temperature coefficient (TC) of photochemical reactions has one of the values  $1.03 \pm 0.03$ ;  $1.20 \pm 0.03$ , or  $1.40 \pm 0.03$ . The reactions in the photographic plate have the first value, whilst all reactions in which one of the halogens plays an important part have the third value. The reactions in the photographic plate are influenced by the wave length of the exciting light thus: ordinary plates with white light have TC 1.06, plates sensitised with Pinachrome have TC for blue light,  $\lambda = 436\mu$ , 1.04; yellow light  $\lambda = 579\mu$ , 1.08, and for green light  $\lambda = 546\mu$ , 1.08; plates which have been previously subjected to very subdued light have TC for blue light 1.03 and for yellow or green light 1.04. (Cf. J.C.S., March.)—J. F. S.

*Photometry; The wedge method of* —. G. I. Higson. Phot. J., 1921, 61, 93—96.

IN the crossed wedge method of photometry first described by Luther (Brit. J. Phot., 1910, 57, 664) the author uses generally for the first exposure either a shallow Goldberg wedge of 0.75 density increase per inch, for examination of the under-exposure portion of the plate, or a steep wedge of 1.9 per inch where the whole curve is required. For the second exposure, through a crossed wedge and negative, a wedge of gradation about 1.8 per inch is employed. A preliminary examination of the spectral selectivity of the wedges was made; it was found that calibration in a visual photometer gave the same result, within 1%, as photographic calibration with white light and a bromide emulsion. It is preferable to make the second exposure on a plate of very steep gradation such as a "process" plate. Both wedges used are scaled and, by selection of the actual exposures given, so that known lines on the scales correspond with definite exposures, the speed of the plate can be read off the final print. The sharpness of the final print is increased by treatment with Farmer's (thiosulphate-ferricyanide) reducer.—B. V. S.

*Transparency of some yellow dyes for ultra-violet light.* F. Stumpf. Z. wiss. Phot., 1921, 20, 183—188.

A TABLE is given showing the transparency regions of solutions of 25 representatives of 11 types of dyestuffs. The most suitable for the preparation of an ultra-violet (transmission) filter are Tartrazine, Flavazine L., Cotton Yellow, Auramine, and Acriline Gold-yellow, which have a transparency region between the limits 350 and 300 $\mu$ . Details are given of the method of determining transparencies photographically, using a quartz spectrograph and a Hartmann microphotometer, and the mathematical expression for the absorption coefficient is deduced. Curves are given showing the change of absorption with wave-length in the ultra-violet region for seven of these dyes at various concentrations.

—B. V. S.

*[Photographic] developers; Exhaustion tests on* —. W. Ermen. Brit. J. Phot., 1921, 68, 64—65.

A NUMBER of prints, which had all received the same exposure behind a Sanger-Shepherd test plate, were developed one after the other in the same lot of developer. The first print of a series was developed practically to finality and the subsequent prints developed to the same amount judging by the density

of the last visible square; the series was continued until this square could not be made visible in a reasonable time or until the time of appearance became unduly long, the time of appearance being taken as a measure of the activity of the developing solution. Number of prints was plotted against time of appearance, the resulting curve showing the method and rate of exhaustion of the developer. *p*-Aminophenol, monomethyl-*p*-aminophenol (metol), dimethyl-*p*-aminophenol, *p*-amino-*o*-cresol (monomet), methylamino-*o*-cresol, and *p*-amino-*m*-cresol were tested as carbonate developers at a strength of 2 g. per l. The curves show appreciable variation in time of appearance and in number of prints obtainable from the same quantity of developer. Quinol and chloroquinol, tested in the same way, but at 4 g. per l., show much longer times of appearance and much quicker rates of exhaustion. Curves are also given for these two developers in caustic alkali solution and in various mixtures with metol and monomet.—B. V. S.

*Sodium silver thiosulphate; Reduction of — by hydrosulphite.* A. Steigmann. *Kolloid Zeits.*, 1921, 28, 29—31.

A discussion of the physical development of the latent photographic image by sodium hydrosulphite in plates and papers after they have been fixed. The silver required for the development is obtained from the silver sodium thiosulphate of the fixing bath.—J. F. S.

[Photographic] silver images; Quinone and other oxidising agents for the conversion of — into bromide or chloride. A. and L. Lumière and A. Seyewetz. *Brit. J. Phot.*, 1921, 68, 6—7.

Quinone and its sulphonic acid may be used in a similar way to various inorganic oxidising agents for the bleaching of silver images. In conjunction with hydrochloric or hydrobromic acid, or other suitable acid and a chloride or bromide, the silver is converted to the halide and the quinone reduced to quinol. The reaction is fairly rapid, there is no staining of the film, and the resulting halide image may be treated by any of the usual methods for reduction, intensification, etc.—B. V. S.

*Colloid chemistry and photography.* Lüppo-Cramer. *Kolloid Zeits.*, 1921, 28, 25—29.

A discussion of the physical and chemical development of the latent image in photographic plates.—J. F. S.

#### PATENTS.

*Coloured photographic pictures; Manufacture of —.* J. H. Christensen. E.P. 133,034, 22.9.19. Conv., 20.9.18.

If a film carrying a silver image and stained with certain dyes is treated with a suitable reducing agent, the dye is bleached in the neighbourhood of the silver image in proportion to the amount of the image, after which the silver may be removed by means of Farmer's reducer. Suitable dyes are Oxamin Fast Rose, Dianil Pure Blue, Aurophenine, Congo Pure Blue, Chicago Blue 6B, etc., mostly dianil dyes. With some dyes an ordinary developer, such as amidol, will reduce the dye, but most of them require a much stronger reducer such as sodium hydrosulphite. A convenient method is to add the dye to the emulsion before coating and after exposure to develop with a strongly restrained hydrosulphite solution which bleaches the dye at the same time. Two or more such coloured sensitive films may be coated on the same base, a multi-coloured image being then produced.—B. V. S.

*Colour photography. Element for use in colour photography.* R. L. Stinchfield, Assr. to Eastman Kodak Co. U.S.P. (A) 1,364,958 and (B) 1,364,959, 11.1.21. Appl. (A) 19.7.19 and (B) 6.2.20.

(A) A support, including a light-reflecting surface, is provided with an alternating series of filter areas and non-filter areas of approximately the same size and with a light-sensitive film yielding an opaque image. (B) The support for the colour-filters and sensitive film is made of opaque, light-absorbent material.—B. V. S.

*Photographic film.* F. W. Lovejoy, Assr. to Eastman Kodak Co. U.S.P. 1,342,590, 8.6.20. Appl., 6.9.19.

THE sensitive layer on a film is protected from the injurious action of moisture by a transparent layer of fatty acid (palmitic or stearic acid), which is saponified and dissolved by the alkali carbonate in the developing bath.

*Celluloid.* S. H. Wood. U.S.P. 1,364,342, 4.1.21. Appl., 16.5.19.

A PLASTIC, non-inflammable, transparent substance suitable for the manufacture of cinematograph films, consists of pyroxylin, naphthalene, ferric chloride, and gelatin.—A. J. H.

*Silver print-out papers; Means for toning —* Akt.-Ges. f. Anilin-Fabr. G.P. 328,559, 13.10.17.

THE toning bath contains in place of the usual gold salt a lead salt along with a salt of copper, cobalt, iron, nickel, manganese, uranium, tungsten, molybdenum, zinc, or bismuth.—B. V. S.

#### XXII.—EXPLOSIVES; MATCHES.

*Nitrocellulose; Analysis of mixed acid for the production of —.* E. Berl and W. von Boltzstern. *Z. angew. Chem.*, 1921, 34, 19—21.

A SCHEME for the analysis of nitrating acid after use is described. The total acidity is determined by titration with alkali, and the nitrogen acids by Arnd's method (J., 1921, 43A). Nitrous acid and total organic matter are determined jointly in terms of  $N/2$  permanganate solution by boiling with an excess of the latter and titrating the excess by means of potassium iodide and thiosulphate solution. Nitrous acid and readily oxidisable organic matter are determined jointly in terms of  $N/2$  permanganate solution by titrating with the latter at 40°—45° C. Total organic matter alone is determined by boiling with permanganate solution as above, after destruction of nitrous acid by boiling the diluted sample with ammonium sulphate or urea. From the data furnished by these operations, which with the exception of Arnd's method are described in detail, the proportions of sulphuric, nitric, nitrous ( $N_2O_3$ ), and oxalic acids, and cellulose products ( $C_6H_{10}O_5$ ) are calculated by formulæ given.—J. H. L.

#### PATENTS.

*Initial priming compositions [containing lead azide]; Manufacture of —.* W. Eschbach. E.P. 156,429, 14.2.20.

IN the manufacture of priming compositions containing lead azide and another constituent, such as lead trinitroresorcinate, the mixing of the ingredients may be accomplished with greater safety if one of them, preferably the more absorptive one, is first treated with a "phlegmatizing" medium, such as benzene. To enable the mixed composition to be pressed satisfactorily, a binding agent such as resin or colophony may be dissolved in the benzene.—W. J. W.



**Powder; Progressively-burning** — and process of making the same. R. G. Woodbridge, jun., K. K. V. Casey, and C. I. B. Henning, Assrs. to E. I. du Pont de Nemours and Co. U.S.P. 1,354,442, 28.9.20. Appl., 25.2.20.

**NITROCELLULOSE** powder, the grains of which are coated with a deterrent (E.P. 127,871 and 127,872; J., 1919, 604 A), is agitated with a mixture of a black powder composition and dinitrotoluene or other non-volatile solvent of nitrocellulose, in order to produce a readily ignitable coating on the grains.

### XXIII.—ANALYSIS.

**Anemometers of great sensitivity, especially applicable to the investigation of slow rates of flow of gases; Thermal effect produced by a slow current of air flowing past a series of fine heated platinum wires, and its application to the construction of hot-wire** — J. S. G. Thomas. Phil. Mag., 1921, 41, 240—258.

**ELEVEN** platinum wires of diam. 0.101 mm. were inserted in a flow tube, in close juxtaposition, transversely to the direction of flow of the air stream in the tube. The plane of the wires was parallel to the direction of flow of the stream, and the wires were heated by a current of 1.1 amp. The change of temperature of the individual wires produced by various velocities of the air stream up to 25 cm. per sec. was investigated, and it was found that the maximum increase of temperature attained a value of about 200° C. in the case of the last wire of the series. The results indicate that when a coil of fine wires is employed for purposes of heating a slow stream of air or other gas, fusion of one or more turns of the coil may result owing to the thermal effect produced in the wires by the heat conducted by the stream, and also that in certain types of electrical gas meters the relative dispositions of the heating coil and platinum thermometers employed are of importance, otherwise anomalous results may be obtained when the velocity of the stream of gas is small. By suitable selection of two or more of the heated wires, the sensitivity of the directional type of hot-wire anemometer previously described (J., 1920, 803 A) may be very considerably increased.—J. S. G. T.

**Melting points and critical temperatures; Simple method for the determination of** — H. Rassow. Z. anorg. Chem., 1920, 114, 117—150.

For the determination of comparatively high melting points and critical temperatures an apparatus was devised consisting essentially of a solid copper cylinder 10 cm. long and 5 cm. diam., bored axially with a hole 6.5 cm. long by 3.5 mm. diam. and transversely through the middle with a hole of the same diam. intersecting the first. The first hole is to take the specimen under examination, the second to enable the specimen to be viewed with a telescope, with the aid of a bright source of light suitably placed to transmit light through the transverse hole to the telescope. The copper cylinder is heated electrically by a winding of nickel wire, the whole being suitably lagged and insulated. The temperature is measured by means of a thermo-couple inserted in a second hole bored close to and parallel with the axial hole and extending half way down the cylinder. The specimen to be examined is sealed in a small narrow tube of hard glass or transparent quartz, which is suspended in the axial hole to come within the field of vision of the telescope through the transverse hole. The apparatus can be used for determinations up to 1080° C. with an accuracy of approximately  $\pm 1^\circ$  C. The following melting points were determined:—KI, 684.1°; KCN,

601.2°; NH<sub>4</sub>Cl, 519.7°; NH<sub>4</sub>Br, 541.9°; NH<sub>4</sub>I, 551°  $\pm$  3° (considerable dissociation occurred); N(CH<sub>3</sub>)<sub>3</sub>Cl, 420°  $\pm$  10°; arsenic, 818°. The apparatus was also used for examining the miscibility of halogen salts of the alkali metals and ammonium. With a specially devised manometer a determination was made of the vapour pressure of ammonium chloride between 340° and 520°; the vapour pressure of NH<sub>4</sub>Cl at its melting point is 34.5 atm.

—E. H. R.

**Standard solutions; Normalities of** — Y. Osaka. Mem. Coll. Sci., Kyoto, 1920, 4, 113—125.

TABLES are given showing the true normality at temperatures from 5° to 30° C. of a number of solutions standardised at 15°, 20°, or 25° C. in glass vessels. The solutions tabulated include oxalic acid (N/1 and N/10), hydrochloric, nitric, and sulphuric acids (N/1), sodium carbonate (N/1 and N/10), sodium hydroxide (N/1), sodium chloride, silver nitrate, and potassium permanganate (N/10 and N/100), and ammonium thiocyanate (N/10). In the case of N/10 and N/100 solutions, the changes of volume with temperature may be taken as equivalent without any greater error than 0.5%.

—E. H. R.

**Iron; Volumetric determination of** — in the presence of a large quantity of hydrochloric acid. Mourice. Ann. Chim. Analyt., 1921, 3, 23—25.

POTASSIUM bromide is added to a ferrous salt solution containing a large proportion of hydrochloric acid, and a current of air is bubbled through the mixture while this is titrated with permanganate solution. The air is then passed through potassium iodide solution containing starch solution; as soon as all the ferrous salt has been oxidised, the next drop of permanganate solution added liberates bromine, which is carried over into the potassium iodide and a blue coloration results.—W. F. S.

**Chromic acid; Iodometric determination of** — I. M. Kolthoff. Z. anal. Chem., 1920, 59, 401—415.

THE conclusions published in a recent paper (J., 1919, 680 A) are confirmed. Potassium bichromate solutions, after addition of potassium iodide, may be accurately titrated at once with thiosulphate solution, provided at least 20 c.c. of 4N hydrochloric acid is added per 100 c.c. of the reaction liquid. Thiosulphate solutions may be standardised, with a possible error of less than 0.1%, by means of potassium bichromate, iodate, or bromate, oxalic acid, iodine, or cyanogen iodide, all of which can be purified without much difficulty. Potassium bichromate, as purchased, commonly contains sulphates and free chromic acid, and may be purified by recrystallising several times from water and dried at 120° C.; it should not be fused over a Bunsen burner.—J. H. L.

See also pages (A) 137, *Coal* (Dolch); *Carbon dioxide in coal* (Sinnatt and Harrison). 138, *Petrol* (Bordas); *Mineral oils* (Armani and Rodano); *Lubricating oils* (Robertshaw). 141, *Water in tar*; *Tar* (Falcicola). 142, *Dihydronaphthalenes* (Straus and Lemmel). 150, *Iron ores* (Leteur); *Carbon in metals* (Bedin). 153, *Soya bean oil* (Dall'Aquila). 157, *Sugar mixtures* (Browne); *Sucrose* (Schrefeld). 158, *Malt* (Roeder, also Matthews); *Sugar in wine* (Fresenius and Grünhut). 159, *Methyl alcohol* (Rahe); *Jard* (Kerr); *Histidine* (Thrun and Trowbridge); *Lead in baking powders* (Patten and Mains). 160, *Hydrogen sulphide in water*. *Starch-iodine reaction* (Chrétien and Vandenberghé). 162, *Eucalyptol* (Bennett and Salamon). 163, *Photometry* (Higson). 164, *Mixed acid* (Berl and von Boltensern).



### Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

#### I.—GENERAL; PLANT; MACHINERY.

##### APPLICATIONS.

Atkinson, and Stein and Atkinson. Continuous furnaces. 4499. Feb. 8.  
Barbet et Fils et Cie. Continuous rectification of liquid air etc. 4898. Feb. 11. (Fr., 11.2.20.)  
Bassler. Drying materials. 4894. Feb. 11.  
Coath. Lubricant. 5448. Feb. 17.  
Deutsche Evaporator A.-G. Kilns. 5094. Feb. 14.  
Greaves. Washing granular, powdered, etc. material. 4674. Feb. 10.  
Herring. Filtering apparatus. 5111. Feb. 14.  
Holland. Separation of materials. 5649. Feb. 18.  
Humphries. Apparatus for drying, cooling, dehydrating, etc. 4781. Feb. 10.  
Ison. Furnaces. 4607. Feb. 9.  
Johnson (Badische Anilin u. Sodafabr.). 5127. See II.  
Kilner. Apparatus for drying solids. 5115. Feb. 14.  
Minton. Lubricants. 5275 and 5445. Feb. 16 and 17.  
Seaman. Grinding and/or mixing mills. 4790. Feb. 10.  
Straatman. Process for decolorising liquids. 5656 and 5765. Feb. 18 and 19. (Holland, 3.12.20, 13.1.21.)  
Tecbno-Chemical Laboratories, Ltd., and Testrup. Drying. 5353. Feb. 16.  
Tecbno-Chemical Laboratories, Ltd., and Testrup. Evaporators etc. 5488. Feb. 17.  
Tecbno-Chemical Laboratories, Ltd., and Testrup. Heating processes. 5489. Feb. 17.

##### COMPLETE SPECIFICATIONS ACCEPTED.

27,519 (1919). Miller, and Fletcher and Co., Ltd. Filters. (158,387.) Feb. 16.  
27,520 (1919). Haines. Filtering apparatus. (158,663.) Feb. 23.  
28,778 (1919). Bedford and Oldroyd. Drying apparatus. (158,734.) Feb. 23.  
30,243 (1919). Carpenter. Collection of gases in chambers. (158,758.) Feb. 23.  
26,025 (1920). McKean and Jones. Filtering apparatus. (158,497.) Feb. 16.  
26,556 (1920). Metcalfe. Continuous kilns of the tunnel type. (158,498.) Feb. 16.

#### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

##### APPLICATIONS.

Beilby. Carbonisation of coal, shale, peat, etc. 5352. Feb. 16.  
Bentley. Gas-generators. 5138. Feb. 14.  
Corthesy. Means for distilling or carbonising pulverulent fuel. 4961. Feb. 12.  
Gartlan and Gooderham. Treatment of petroleum oils. 4496—7. Feb. 8.  
Helms. Gas manufacture. 5573. Feb. 18.  
Johnson (Badische Anilin u. Sodafabr.). Process for purifying gases. 5127. Feb. 14.  
Lush. Treatment of producer gas etc. 4980. Feb. 12.  
MacLaurin. Producer for dealing with low-grade fuels. 4337. Feb. 7.  
Roberts. Recovering by-products from distillate gas. 4996. Feb. 12.  
Rollason. Carbonisation of coal. 5035. Feb. 14.

Simpson. Apparatus for gasifying fuel. 4490. Feb. 8.

Szarvasy. Graphitising pre-formed carbon bodies. 4375. Feb. 7. (Hungary, 18.7.18.)

Szarvasy. Production of fine soot. 4376. Feb. 7. (Hungary, 18.7.18.)

Szarvasy. Manufacture of pure retort carbon. 4378. Feb. 7. (Hungary, 28.2.14.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

12,134 (1917). Jones and MacDonald. Vertical retorts for destructive distillation of coal, shale, etc. (109,800.) Feb. 23.  
18,252 (1919). Soc. de Fours à Coke. Coke ovens. (130,974.) Feb. 16.  
21,969 (1919). Scrivner. Suction gas generators. (132,269.) Feb. 16.  
27,090 (1919). Bale. Apparatus for carbonising coal, lignite, shale, etc. (158,622.) Feb. 23.  
27,290 (1919). Brownlee and Uhlinger. Combustible gaseous compositions, especially for use in cutting, welding, etc. (158,633.) Feb. 26.  
27,585 (1919). Duncombe and Singer. Fuel. (158,671.) Feb. 23.  
27,770 (1919). Perry. Apparatus for distilling carbonaceous material. (158,394.) Feb. 16.  
28,072 (1919). Naaml. Vennoots. Nederlandsche Licht Olie Maatsch. Converting higher molecular hydrocarbons into lower molecular ones. (135,197.) Feb. 23.  
28,253 (1919). White. Generators for producer-gas plants. (158,409.) Feb. 16.  
30,117 (1919). Lowe. Plant for making blue water gas in conjunction with coal gas. (149,928.) Feb. 23.

#### III.—TAR AND TAR PRODUCTS.

##### APPLICATIONS.

Atack. 4556. See XX.  
Atack. Oxidation of hydrocarbons. 4557. Feb. 9.  
Atack. Manufacture of anthraquinone or its derivatives. 5672. Feb. 19.  
Bell Bros., Ltd., Lowe, and Roelofsen. Treating acid tar from washing benzol etc. for recovery of sulphuric acid etc. 5647. Feb. 18.  
Chem. Engineering and Wilton's Patent Furnace Co., Ltd., Wilton, and Wilton. Distillation of tar. 4902. Feb. 11.  
Glysyn Corp. Chlorination of hydrocarbons. 5665. Feb. 18. (U.S., 31.8.20.)

##### COMPLETE SPECIFICATION ACCEPTED.

26,759 (1919). Anderson and Meikle. Treatment of coal tar. (158,337.) Feb. 16.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

##### APPLICATIONS.

Dreyfus. Manufacture of cellulose derivatives. 4839. Feb. 9.  
Kampf. Recovery of carbon disulphide in working up viscose. 4523. Feb. 8. (Ger., 28.10.20.)  
Müller. Refining wood cellulose for manufacturing a cotton-like fibre. 4539. Feb. 8.  
Stevenson. Manufacture of artificial silk from acetylcellulose. 5044. Feb. 14.  
Tibuzzi. Manufacture of paper. 5372. Feb. 16. (Ital., 24.7.20.)  
Zdanowicz. Manufacture of artificial silk, horsehair, etc. from cellulose acetates. 5318. Feb. 16.  
Zdanowicz. Manufacture of films, sheets, etc. from cellulose acetates. 5319. Feb. 16.

##### COMPLETE SPECIFICATIONS ACCEPTED.

17,082 (1919). Flaherty. Pyroxylin solvent and pyroxylin compositions. (158,586.) Feb. 23.  
25,494 (1919). Du Pont de Nemours and Co. Pyroxylin compositions. (153,972.) Feb. 16.

28,191 (1919). Glanzfäden A.-G. Manufacture of a wool substitute from cellulose solutions. (135,206.) Feb. 23.  
12,106 (1920). Summers. Process of retting flax etc. (158,807.) Feb. 23.

#### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

##### APPLICATIONS.

Clavel. Treatment of union or mixed fabrics. 4638. Feb. 9.  
Farrell. Dye-jigs or becks. 4956. Feb. 12.  
Frei. Apparatus for treating hanks of yarn with liquid. 4912. Feb. 11. (Switz., 12.2.20.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

26,801 (1919). British Cellulose and Chem. Manuf. Co., Briggs, and Palmer. Dyeing or colouring fibres, threads, or fabrics. (158,340.) Feb. 16.  
28,230 (1919). Calico Printers' Assoc., Ashton, and Mellor. Overprinting fabrics and yarns. (158,407.) Feb. 16.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

##### APPLICATIONS.

American Zeolite Corp. Preparation of artificial base-exchanging bodies. 5615. Feb. 18. (U.S., 19.2.20.)  
Armour Fertilizer Works. Production of aluminium chloride. 4895. Feb. 11. (U.S., 24.3.20.)  
Barbet et Fils et Cie. 4898. See I.  
Bassett. 5752. See X.  
Bassett. Manufacture of alkali and alkaline-earth sulphides. 5753. Feb. 19. (Fr., 20.2.20.)  
Bell Bros., Ltd., and others. 5647. See III.  
Brat. Recovery of nitrogen from peat etc. 5518—9. Feb. 17. (Ger., 18.2.20.)  
Cumberland Coal Power and Chemicals, Ltd., Jaques, and West. Production of hydrogen. 4642. Feb. 9.  
Deutsche Gold- u. Silber-Scheide-Anstalt, and Liebknecht. Manufacture of prussic acid. 4419. Feb. 7.  
Dutt and Dutt. Preparation of titanium dioxide and alumina from bauxite. 5626. Feb. 18.  
Griffith. 5669. See XXI.  
Guignard. Extraction of nitrogen from nitrogen compounds of titanium. 5108. Feb. 14. (Fr., 19.3.20.)  
Williams. Preparation of water-soluble phosphates. 4672. Feb. 10.

##### COMPLETE SPECIFICATIONS ACCEPTED.

15,764 (1918) and 395 (1919). Soc. Indus. de Prod. Chimiques. Separation of sodium and ammonium sulphates from the double salt. (128,895 and 158,282.) Feb. 16.  
19,027 (1919). Macleod (Browning). Production of hydrogen sulphide. (158,288.) Feb. 16.  
21,220 (1919). Matheson. Utilisation of alunite ore. (158,293.) Feb. 16.  
29,030 (1919). Wang. Manufacture of ammonium perchlorate. (137,034.) Feb. 23.  
31,816 (1919). Welter. Production of soda containing water of crystallisation. (136,841.) Feb. 23.  
1012 (1920). Frischer. Concentration of dilute nitric acid. (137,834.) Feb. 23.  
12,751 (1920). Terrisse and Levy. See XVI.

#### VIII.—GLASS; CERAMICS.

##### APPLICATIONS.

Bailey. Kilns for firing pottery etc. 4974. Feb. 12.  
Blythe. Brick, pottery, etc. kilns. 5399. Feb. 16.

#### IX.—BUILDING MATERIALS.

##### APPLICATION.

Passmore. Manufacture of fireproof or insulating material. 5742. Feb. 19.

##### COMPLETE SPECIFICATIONS ACCEPTED.

27,607 (1919). Crosbie. Asphaltic cement or bituminous composition for road making, etc. (158,674.) Feb. 23.  
27,647 (1919). Baylor. Slow-setting cement and method of producing same. (158,390.) Feb. 16.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

##### APPLICATIONS.

A.-G. Brown, Boveri et Cie. Electrically-heated muffle furnaces. 5614. Feb. 18. (Switz., 18.2.20.)  
Amalgamated Zinc (De Bavay's), Ltd. Recovery of lead and silver from sulphide ores etc. 4913. Feb. 11. (Australia, 13.2.20.)  
Ashcroft. Metallurgy of metal-bearing ores or products. 5128. Feb. 14.  
Bassett. Extraction of metals or metalloids from their oxides. 5752. Feb. 19. (Fr., 20.2.20.)  
British Thomson-Houston Co. (General Electric Co.). Bearing material. 4512. Feb. 8.  
British Thomson-Houston Co. (General Electric Co.). Electrodeposition of metals. 5112. Feb. 14.  
Campbell, Gifford, and Waite. Electric furnaces for non-ferrous metals. 4432. Feb. 7.  
Diepschlag. Working blast-furnaces etc. 4922. Feb. 11.  
Diepschlag. Making ready fuel and smelting charges. 4923. Feb. 11.  
France. Washing fine particles of minerals. 4388. Feb. 7.  
Haglund. Treatment of copper-nickel matte. 4360. Feb. 7. (Norway, 10.2.20.)  
Jackson (Soc. Metalurgica Chilena Caprum). Treatment of ores. 4782. Feb. 10.  
Jackson (Leadizing Co.). Coating iron or steel articles with lead. 5759. Feb. 19.  
Jones, and Minerals Separation, Ltd. Recovery of flotation agents from flotation products. 4911. Feb. 11.  
Nettleton. Separating minerals etc. 4718. Feb. 10.  
Perkins. Treatment of oxidised ores of copper. 5068. Feb. 14.  
Rheinisch-Nassauische Bergwerks- u. Hütten A.-G., and Spieker. Extracting zinc from lead slags, zinc-retort residues, poor zinc ores, etc. 5472. Feb. 17. (Ger., 17.3.20.)  
Stevens. Reduction of ores. 5120. Feb. 14. (U.S., 26.3.20.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

26,470 (1919). Chikashige and Uno. Recovering selenium and noble metals from electrolytic alimes etc. (134,536.) Feb. 16.  
26,530 and 26,633 (1919). Middlemiss. Treatment of finely-crushed ores etc. (158,320.) Feb. 16.  
26,623 (1919). Ballantine. Production of rustless articles of steel. (158,329.) Feb. 16.  
27,290 (1919). Brownlee and Uhlinger. See II.  
29,031 (1919). Heskett. Manufacture of metal powders. (158,740.) Feb. 23.  
30,960 (1919). British and Foreign Chemical Producers, Ltd. (Rhein. Kampferfabr.). Pickling iron or steel. (158,768.) Feb. 23.  
3676 (1920). Schwartz. Silver-plating powder. (158,460.) Feb. 16.  
12,372 (1920). Soncini. See XI.

#### XI.—ELECTRO-CHEMISTRY.

##### APPLICATIONS.

British Thomson-Houston Co. 5112. See X.  
Campbell, Gifford, and Waite. Graphitising-furnaces. 4431. Feb. 7.

Campbell and others. 4432. *See X*  
Deutsche Gold- u. Silber-Scheide-Anstalt, and  
Liebknecht. Platinum anodes. 4420. Feb. 7.  
Hiorth. Induction furnaces. 5501. Feb. 17.  
(Norway, 21.2.20.)  
Michel. Manufacture of electrodes. 4858.  
Feb. 11.  
Szarvasy. 4375. *See II*.  
Szarvasy. Manufacture of carbon electrodes.  
4377. Feb. 7. (Hungary, 29.11.17.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

26,470 (1919). Chikashige and Uno. *See X*.  
28,772 (1919). Powell. Primary batteries.  
(158,733.) Feb. 23.  
8444 (1920). Soc. Metallurgique du Frayol.  
Electric furnaces. (157,051.) Feb. 16.  
12,372 (1920). Soncini. Electric crucible  
furnaces. (142,836.) Feb. 16.

#### XII.—FATS; OILS; WAXES.

##### APPLICATIONS.

Dutt. Treatment and decolorisation of vegetable  
oils and glycerin. 4613. Feb. 9.  
White (Safetee Soap Corp.). Cocoa-butter com-  
positions. 5668. Feb. 18.

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,222 (1919). Barbet et Fils et Cie. Continuous  
distillation of glycerin from residue obtained in dis-  
tilling fermented liquors. (129,649.) Feb. 23.  
19,883 (1920). Rogers. Extraction of oils from  
fatty substances. (147,834.) Feb. 23.

#### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

##### APPLICATION.

Bakelite Ges., and Hessen. Manufacture of con-  
densation products from phenols and aldehydes.  
5237. Feb. 15. (Ger., 1.3.20.)

#### COMPLETE SPECIFICATION ACCEPTED.

31,393 (1919). Vickers, Ltd., Ioco Rubber and  
Waterproofing Co., Ltd., and Nuttall. Condensa-  
tion of phenolic bodies with aldehydic compounds.  
(158,447.) Feb. 16.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

##### APPLICATION.

Davidson. Coagulant for rubber latex. 5401.  
Feb. 16.

#### COMPLETE SPECIFICATION ACCEPTED.

32,259 (1919). Smith. Process of devulcanising  
rubber. (158,783.) Feb. 23.

#### XV.—LEATHER; BONE; HORN; GLUE.

##### APPLICATION.

Manvers. Preparation of hides for tanning.  
5677. Feb. 19.

#### XVI.—SOILS; FERTILISERS.

##### APPLICATIONS.

Haegge. Production of fertilisers containing phos-  
phoric acid and potash. 5195. Feb. 15.  
Williams. 4872. *See VII*.

#### COMPLETE SPECIFICATION ACCEPTED.

22,020 (1920). Tavrogas, Roche, and Martin.  
Treatment of whey produced in cheese manufacture  
and production of nitrogenous matter applicable  
as a fertiliser. (158,816.) Feb. 23.

#### XVII.—SUGARS; STARCHES; GUMS.

##### COMPLETE SPECIFICATION ACCEPTED.

12,751 (1920). Terrisse and Levy. Regenerating  
hydrochloric acid used in the manufacture of  
glucose. (154,170.) Feb. 18.

#### XVIII.—FERMENTATION INDUSTRIES.

##### APPLICATIONS.

Briacoe. Malting. 5556. Feb. 18.  
Gilmour. Manufacture of yeast. 5444. Feb. 17.

#### COMPLETE SPECIFICATION ACCEPTED.

17,222 (1919). Barbet et Fils et Cie. *See XII*.

#### XIX.—FOODS; WATER PURIFICATION; SANITATION.

##### APPLICATIONS.

American Zeolite Corp. 5615. *See VII*.  
Dunham. Production of casein etc. 5065.  
Feb. 12.  
White. 5668. *See XII*.

#### COMPLETE SPECIFICATIONS ACCEPTED.

11,080 (1917). Paterson. Sterilising liquids.  
(158,578.) Feb. 23.  
27,019 (1919). Rushton. Softening or purifica-  
tion of water. (158,620.) Feb. 23.  
27,773 and 30,595 (1919) and 19,605 (1920).  
Townsend. Manufacture of articles of food.  
(158,634.) Feb. 23.  
14,624 (1920). Sgalitzer. Manufacture of a food  
preparation from blood. (143,919.) Feb. 16.  
22,020 (1920). Tavrogas and others. *See XVI*.  
26,223 (1920). Magrath. Treatment of water for  
softening, sterilising, etc. (158,498.) Feb. 16.

#### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

##### APPLICATIONS.

Atack. Condensation of *o*-benzoylbenzoic acid or  
its derivatives. 4556. Feb. 9.  
Bader, and British Cellulose and Chem. Manuf.  
Co. Manufacture of acetic anhydride etc. 4637.  
Feb. 9.  
Badische Anilin u. Sodafabrik. Manufacture of  
alcohol. 4634. Feb. 9. (Ger., 9.2.20.)  
Glysyn Corp. 5665. *See III*.  
Soc. Chim. Usines du Rhône. Manufacture of  
saccharin. 5019. Feb. 12. (Ger., 25.6.20.)  
Zinke. Manufacture of perylene. 5103. Feb. 14.  
(Austria, 2.7.20.)  
Zinke. Manufacture of dioxyperylene. 5104.  
Feb. 14. (Austria, 2.7.20.)

#### COMPLETE SPECIFICATION ACCEPTED.

21,371 (1920). Henning. Non-flammable volatile  
liquid. (158,494.) Feb. 16.

#### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

##### APPLICATION.

Griffith. Schlippe's salts etc. 5669. Feb. 19.

#### COMPLETE SPECIFICATION ACCEPTED.

27,574 (1919). Camiller and Hay. Multicolour  
screens for colour cinematography and photo-  
graphy. (153,670.) Feb. 23.

#### XXIII.—ANALYSIS.

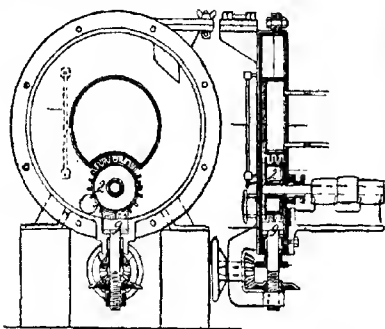
##### APPLICATION.

Andrews, and Victoria Falls and Transvaal Power  
Co. Quantitative detection of carbon dioxide and  
combustible gases containing carbon. 5654. Feb. 18.

**I.—GENERAL; PLANT; MACHINERY.**

*Colloid mill and its application to chemical technology.* B. Block. Verein deutsch. Chem., 11.9.20. Z. angew. Chem., 1921, 34, 25—30. (Cf. J., 1920, 539 A.)

THE physical conditions governing the production of disperse phases are briefly reviewed and various types of mills hitherto available for the production



of such systems are described. The so-called colloid mill (see fig.) comprises a drum, 2, provided with a number of arms rotating in a chamber containing the substance to be dispersed and the dispersing medium. The liquid is projected by the rotating arms against obstructions, 9, and dispersion of the substance results. The principal difficulties in the operation of the mill consist in rendering the axle-packing tight and in attaining a sufficiently high circumferential velocity of the drum. In the case of a colloid mill employed for dispersing cellulose this material was reduced to particles of the order of 0.0003 mm. when the circumferential velocity of the drum was 20 m. per sec., whilst when the circumferential velocity was 30 m. per sec. the size of the particles was reduced to 0.0001 mm., and the true colloidal condition was attained when the velocity was 40 m. per sec. Smaller mills, with which circumferential velocities up to 500 m. per sec. may be attained, are contemplated. The development of such mills depends upon the discovery of a steel of extremely high tenacity. By employing two drums rotating in opposite directions the necessary circumferential velocity can be reduced. The operation of the mill can also be expedited by employing suitable accelerators of the dispersion process. The lay-out of installations for the production of colloidal cellulose for the manufacture of insulating materials etc. and of colloidal shale for the separation of the oil is detailed.

—J. S. G. T.

*Alinement charts: Construction and use of —* A. J. V. Umanski. Chem. Trade J., 1921, 68, 233—236, 269—272.

An account of the methods of nomography and their application to cases such as the adiabatic compression of gases, evaporation of solvents, reducing gaseous volumes to normal temperature and pressure, condensing coils and heating plant, the design of crystallising tanks, and heat transmission between moving liquids.

**PATENTS.**

*Coal, minerals, or the like; Washers for classifying —* L. Malécot. E.P. 145,444, 18.6.20. Conv., 18.6.18.

A VESSEL with vertical sides is provided with an offset portion at the top, having an inclined outer

wall and communicating with the main vessel through an opening in the vertical wall which forms a partition between the two portions. The material is fed from a hopper on to the inclined wall and passes downwards through still water into an upward current in the vertical vessel. The heavier portion sinks to the bottom of this vessel, and the lighter portion is carried upward by the water and thence along a horizontal channel on the side of the vertical vessel opposite to that at which the material is admitted.—W. F. F.

*Centrifugal pumps for acids.* Chem. Fahr. vorm. Weiler-ter Meer. E.P. (A) 145,803 and (B) 146,409, 2.7.20. Conv., 28.9.18 and 19.11.17.

(A) A PUMP has a vertical spindle surrounded by a tubular acid trap and carried to a considerable height above the guide-box, the top of the tubular trap being approximately closed by a collar or the like. The head of the inflowing acid must be sufficient to cause the acid to rise through the guide-box into the trap and yet not sufficient to cause the acid to overflow above the trap when the pump is at rest. The pump is designed so that all interior parts may be machined, so that it can be made of, e.g., wrought iron. (B) To prevent the action of the pump emptying the trap, a passage or pipe is provided connecting the trap directly with the inlet pipe.—B. M. V.

*Furnace; Heat-treating —* C. J. Kirk. E.P. 155,712, 24.12.19.

A CHAMBER to receive the goods under treatment is divided into two compartments by a longitudinal wall in which openings are left at top and bottom for circulation of air. Heat is applied by means of flues in the walls and arch of the furnace. Between the flues and chamber is a space through which the air from the chamber can circulate. This space may conveniently be formed by building the inner wall of the side flues of hollow tiles which are open at top and bottom to the chamber.—B. M. V.

*Furnaces; Rotary —* F. Derneden. E.P. 156,835, 26.6.19.

A ROTARY furnace is provided with a number of longitudinal channels surrounding the axis of rotation; the channels are connected together, and also to an axial passage if desired, by means of inclined passages, so that the material passes from one longitudinal channel to the next upon rotation of the furnace. The outer channels, but not the axial passage, may be subdivided by cross partitions.—B. M. V.

*Surface combustion furnace.* F. J. Denk. U.S.P. 1,365,769, 18.1.21. Appl., 5.12.19.

THE space to be heated is surrounded by spaced refractory material divided into a number of sections. Combustible gas is supplied separately to each section, and means are provided for drawing combustion products away from contact with the articles which are being heated.—W. F. F.

*Tunnel kiln.* F. K. Meiser. G.P. 327,089, 15.7.19. Addn. to 319,440 and 320,800 (J., 1920, 475 A, 647 A).

THE crucibles are arranged in a circle and connected by fixed tubes provided with valves. The gas is led into the crucibles through flexible tubes or by means of a circular channel.—L. A. C.

*Pulverising apparatus.* W. L. McLaughlin. E.P. 156,852, 28.8.19.

A ROD-MILL is provided with grinding rods of about the same length as the mill, but of several different diameters, so that the smaller rods enter the interstices between the larger and render the mill

suitable for fine grinding, e.g., of coal. The material is fed evenly over the whole length of the mill by means of a fixed hollow shaft or casing provided with spiral slots and a spiral conveyor. The material that is sufficiently ground is kept in suspension by a series of high-pressure air jets within the mill and removed through one end of the mill by a low-pressure suction.—B. M. V.

*Grinding mill.* H. W. Hardinge. U.S.P. 1,366,651, 25.1.21. Appl., 22.10.19.

A GRINDING mill has, at its outlet end, a classifying compartment shaped as a truncated cone and containing loose tapering rods with their small ends towards the outlet, which is situated at the small end of the outer conical drum.—B. M. V.

*Centrifugal machine.* P. T. Sharples. E.P. 157,688, 22.6.20.

IN centrifugal separators in which a carrier liquid is used to prevent the separated material from sticking to the walls of the basket, a diaphragm is provided near the bottom of the basket, and extending nearly to its walls. The inlet pipe is in the form of a double conduit, the carrier liquid being supplied through the duct below the diaphragm, whilst the pulp to be separated is supplied through the other duct above the diaphragm.—B. M. V.

*Emulsions and the like [e.g., wool washing effluent]; Treatment of —.* W. Broadbridge, E. Edeer, and Minerals Separation, Ltd. E.P. 157,490, 18.7.19 and 12.8.19.

THE emulsion (e.g., wool washing effluent) is subjected to aeration and agitation by means of mechanical beaters which produce a vortex and violent turbulence, the liquid passing through the zone of turbulence several times, and a portion when completely aerated rising through a grid or other perforated division to a zone of comparative quiet where the floating values are removed. Frothing and modifying agents may be added and the process worked at an elevated temperature.—B. M. V.

*Specific gravity of flowing liquid; Apparatus for indicating and recording —.* S. D. Wells and R. J. Marx. E.P. 158,151, 21.1.20.

A BULB with inlet and outlet tubes, the latter arranged above the former, forms one arm of a balance, the other arm being provided with an adjustable counter weight. A portion of the liquid of which the density is to be measured flows (without mechanical connexion) into a funnel on the inlet tube, which is substantially on the pivotal axis, and is discharged through a vented goose neck to an outlet similarly situated.—B. M. V.

*Reaction spaces; Filling material for —.* A. M. Fairlie. U.S.P. 1,365,671, 18.1.21. Appl., 23.3.18.

REACTION spaces are filled with hollow blocks open at both ends, each containing at least one complete turn of a helical vane supported by an axial rod.—W. F. F.

*Dryers; Method of and apparatus for recovering heat from —.* J. O. Ross, Assr. to B. F. Sturtevant Co. U.S.P. 1,365,790, 18.1.21. Appl., 7.3.18.

HOT moist air from a dryer is mixed with atmospheric air at a substantially lower temperature, and the condensed water removed, leaving the air saturated at the lower temperature. The air is then re-heated for drying purposes.—W. F. F.

*Drying gas; Apparatus for recovering heat and solid particles in suspension from a —.* R. G. Brindle and A. H. Flint, Assrs. to Corn Products Refining Co. U.S.P. 1,366,712, 25.1.21. Appl., 3.7.18.

PART of a liquid (which is to be evaporated by contact with a heated drying-gas) is sprinkled into a chamber through which is also passed the drying-gas from the evaporating chamber, which may be carrying particles of the dried liquid in suspension. The liquid after it has collected the heat and dust is returned to the original supply tank. The chamber may be provided with baffles over which the liquid trickles, or other means of increasing the surface of the absorbing liquid.—B. M. V.

*Drying ovens; Process and apparatus for preventing fires in direct-fired —.* P. Schneider. G.P. 326,403, 30.11.17.

STEAM as well as air is mixed with the furnace gases, whereby sparks are extinguished, and improvement effected in the quality of the dried materials.—J. S. G. T.

*Drying apparatus.* O. Pfeiffer. G.P. 327,731, 20.8.18.

AN apparatus for drying loose material comprises a number of drying chambers connected with chests for the supply and withdrawal of the air employed for drying, the whole being mounted so that it can be rotated about a fixed horizontal axis. The charging appliance may be arranged immediately above the drying apparatus, which may be emptied into a chamber immediately underneath. During the drying operation the partitions separating the drying chambers are in a horizontal position, whilst for discharging purposes the apparatus is rotated until the partitions are vertical.—J. S. G. T.

*Volatile solvents; Process of recovering —.* S. S. Sadler. U.S.P. 1,365,791, 18.1.21. Appl., 18.5.20.

THE vapour of the solvent is absorbed by a mixture of sperm oil, lard oil, petroleum oil, and alcohol.—W. F. F.

*Filter.* J. P. Winters and M. McCamon. U.S.P. 1,366,143, 18.1.21. Appl., 23.4.20.

A CLOSED tank is provided with a supply pipe extending downwards nearly to the bottom, and an inverted cone is secured at its apex to the bottom of the pipe with its rim spaced from the wall of the tank. A horizontal zone of filtering material is arranged above the cone. There is an outlet valve just below the rim of the cone and an overflow above the zone of filtering material.—W. F. F.

*Acid receptacles; Outlet for —.* F. W. de Jahn, Assr. to General Chemical Co. U.S.P. 1,366,170, 18.1.21. Appl., 6.5.18.

A RECEPTACLE having an acid-proof lining within a corrodible outer casing is provided with an outlet pipe attached to the casing and having an extension extending into the refractory lining but spaced therefrom and with acid-proof packing in the space.—B. M. V.

*Steam generator.* H. A. Nicholson, Assr. to E. A. Craig. U.S.P. 1,366,207, 18.1.21. Appl. 7.1.18.

A GAS producer is surrounded at one part by a water-jacket, and this in turn by a combustion chamber and then by a feed water coil. Feed water is passed through the coil to the water-jacket and thence to a battery of pipes in the combustion chamber. The pipes communicate with a steam chamber from which steam may be withdrawn.—W. F. F.

*Crystallising receptacle.* F. Merz. U.S.P. 1,366,474, 25.1.21. Appl., 24.2.20.

THE walls of a crystallising vessel are made of flexible material so that crystals may be readily detached.—B. M. V.

*Carrying out chemical reactions in an artificial atmosphere; Process and plant for —.* M. C. J. E. de Loisy. U.S.P. 1,366,720, 25.1.21. Appl., 26.6.19.

THE operations are conducted in a furnace which is open but which is within a closed chamber containing the required gas.—B. M. V.

*Scraper and agitator; Combined —.* A. E. Flowers, Assr. to National Aniline and Chemical Co., Inc. U.S.P. 1,366,777, 25.1.21. Appl., 8.1.20.

A VESSEL of circular section is provided with a rotary scraper and agitator. The actuating arms extend nearly to the walls of the vessel and carry at the ends pivoted scrapers; the main part of each scraper trails behind the end of the arm, but the forward part is in the form of a knife, inclined forwards and outwards, which removes material from the wall of the vessel.—B. M. V.

*Heat-treating bodies; Process of —.* N. H. Fooks. U.S.P. 1,366,778, 25.1.21. Appl., 17.7.20.

A VESSEL of pressure-resisting vessel contains a fixed gas and a liquid. The temperature of the vessel is maintained above the boiling point of the liquid at normal pressure but below the boiling point at the pressure used. The articles to be treated are inserted, treated, and removed while substantially maintaining the required pressure and temperature.—B. M. V.

*High-pressure-air refrigerating machine.* Ges. für Lindes Eismaschinen A.-G. G.P. 323,950, 16.1.18.

AIR is compressed under a very high pressure and then expanded to a lower pressure, but still above atmospheric pressure, say 50 atm., whereby the efficiency of the refrigerating process is increased on account of the disproportionately higher value of the specific heat of air at the higher pressures. For each step down in temperature an additional expansion cylinder with compressor and refrigerating appliance is employed. In many cases the machine can compete successfully with modern types of refrigerating machines employing liquid carbon dioxide, ammonia, etc. as refrigerating agent. The machine is especially applicable for use in connexion with chemical processes carried out in an enclosed space, in mines and under water.—J. S. G. T.

*Filter for purification of gases.* Deutsche Luftfilter-Bauges. m.h.H. G.P. 325,782, 10.10.17.

A FILTER frame in the form of a cage is built up of a number of layers of filtering material supported on separate screens, individually removable or replaceable. Pockets on the gas inlet side of the filter open downwards, while those on the gas exit side open upwards. A number of cages may be held together by means of an angle iron, and the whole made to revolve about a common axis.—J. S. G. T.

*Purification of gases; Plant for electrical — employing perforated electrodes.* Siemens Schuckertwerke G.m.b.H. G.P. 325,956, 4.6.18.

THE collecting space for material precipitated from the gas is covered by the precipitating electrodes, above which the high-tension electrodes are disposed both transversely to and along the direction of flow of the gas stream. In this manner, precipitation is effected both by gravity and the electrical field.—J. S. G. T.

*Purification of gases; Multiple-stage liquefaction process for the —.* Ges. für Lindes Eismaschinen A.-G. G.P. 327,127, 26.3.19.

THE gas to be purified by liquefaction is first cooled at a moderate pressure by the evaporation and expansion of a part of the gas already liquefied, and both portions are subsequently mixed, compressed, and liquefied under high pressure. Purification is effected during the stages of the cooling process.—J. S. G. T.

*Evaporator with chain stirrer.* Gebr. Heine. G.P. 325,799, 16.6.18.

AN evaporator in the form of a bowl or trough is provided with a stirrer consisting of a horizontal frame formed of rods, parallel to the axis of rotation, from which the chains hang freely. The chains cut through the froth on the surface of the liquid, whereby the bubbles burst and the evaporating surface is constantly renewed.—J. S. G. T.

*Evaporation of liquids; Frothing process for the —.* Wo. Ostwald. G.P. 327,976, 30.11.18.

TO facilitate the production of a fine mist, substances are added to the liquid which reduce its surface tension. Frothing may be effected by the introduction of a gas or by the vapours of low-boiling liquids or, if desired, temperature and pressure conditions may be adjusted so that the lightest constituent of the liquid itself is vaporised and acts as the frothing agent.—J. S. G. T.

*Converting liquids or pasty masses [e.g., glycerin fermentation residues] into a dry and easily extractable condition; Process for —.* K. Ebers. G.P. 326,728, 16.12.17.

A DISTRIBUTING medium, insoluble in the extracting medium to be employed, is added to the liquid or pasty mass, and the mixture atomised in a stream of dry air. The residue is obtained in an easily extractable form. In applying the process to the treatment of the residues remaining after the separation of the hulk of the glycerin from glycerin fermentation masses, common salt or any salt insoluble in alcohol or ether is employed as distributing medium, and the dried mixture is extracted with alcohol or ether to recover the remainder of the glycerin. Slime and aluminous substances are by this process converted into forms which do not affect the extraction of the dried residue.—J. S. G. T.

*Cooling or heating systems.* Maschinenfabrik Esslingen. G.P. 327,200, 19.9.18.

A SYSTEM of cooling or heating pipes is built up of a number of U-shaped members, and so can be made of cast acid-resisting silicon-iron. A lateral branch pipe of smaller cross-section opens from the bottom of each member, enabling deposits and condensed liquid to be removed from the system.—J. S. G. T.

*Metallic body to be enclosed in a vessel wherein a high vacuum is to be maintained at a high temperature; Production of a —.* Schott u. Gen. G.P. 330,572, 18.4.18.

THE body is constituted of a metal which has been melted in *vacuo*, so that no further gases or impurities are evolved on subsequent heating.—J. S. G. T.

*Sedimentation apparatus.* The Dorr Co., Assecs. of C. L. Peck. E.P. 133,716, 11.10.19. Conv., 5.10.18.

SEE U.S.P. 1,337,094—5 of 1920; J., 1920, 393 A.

*Dryers; Centrifugal —.* G. H. Elmore. E.P. 158,152, 9.2.20.

SEE U.S.P. 1,342,743 of 1920; J., 1920, 538 A.

*Mixing machines [for concrete etc.]; Mechanical —.* H. Martinez. E.P. 157,557, 20.10.19.

**IIA.—FUEL; GAS; MINERAL OILS AND WAXES.**

*Coal; Formation and chemical constitution of —.*  
F. Fischer and H. Schrader. *Brennstoff-Chem.*, 1921, 2, 37–45.

THE authors conclude from their researches that the cellulose disappeared from the plant substances from which the coal was formed, being decomposed chiefly by bacterial action, and the coal was formed by the conversion of the lignin into humic substances, the percentage of wax and resin increasing owing to the disappearance of the cellulose. The phenols obtained in low-temperature tar are formed by the decomposition of these humic acids in distillation, and the hydrocarbons from the bitumen. (Cf. J.C.S., Mar.)—W. P.

*Gas purification [from sulphur]; Alkalinity tests in —.* C. H. Stone. *Amer. Gas Assoc. Gas J.*, 1921, 153, 341–342.

THE effect of the addition of lime to natural oxide of iron in a purifier is not to increase the absorptive power for sulphur, but to increase the speed of absorption. Calcium or magnesium carbonate is as effective as calcium hydroxide. Their efficacy is due to neutralisation of the sulphuric and thiocyanic acids formed. 2% of calcium carbonate gave the best result. Fouled oxide allowed to stand out of contact with air cannot be revived, owing to the formation of iron disulphide. The absorption velocity in a purifier increases with the temperature, but the practical limit is reached at about 40° C. owing to the risk of the heat of reaction liberating sulphur compounds. Ferrous sulphate and sulphur do not affect the action of oxide except by dilution.—O. I.

*[Lubricating] oils; Cold test apparatus for —.*  
G. H. P. Lichthardt. *J. Ind. Eng. Chem.*, 1921, 13, 145–146. (Cf. Martens, J., 1890, 772.)

THE oil to be tested is placed in a glass tube fitted in an inclined position through a tank containing a freezing mixture (acetone and carbon dioxide snow); the lower end of the tube is bent upwards and connected with an air supply of constant pressure. When the oil becomes solid and does not move under an air pressure of 16 in. of water, the temperature is allowed to rise slowly; the temperature at which the oil appears in the part of the tube protruding from the tank is taken as the "cold test," i.e., the lowest temperature at which the oil will flow.—W. P. S.

*Detecting fire-damp etc.* Erlwein and Becker. See XI.

*Gas analysis.* Möller. See XXIII.

*Flue gas.* Milligan and others. See XXIII.

**PATENTS.**

*Briquette manufacture.* J. F. Lovejoy. U.S.P. 1,366,091, 18.1.21. Appl., 15.10.19.

THE briquettes travel downwards through a zigzag passage and furnace gas passes upwards over the lower portion of the passage to carbonise the briquettes. The gas then passes through perforations in the walls into the upper portion of the passage to preheat the descending briquettes by direct contact therewith.—W. F. F.

*Coal briquettes; Manufacture of coherent — without the use of foreign binding material.* O. Dobbelstein. G.P. 329,054, 25.3.14.

HARD coal dust is mixed with gas-coal dust or sludge, and the mixture briquetted under a pressure of 3000–4000 atm.—B. V. S.

*Crude-oil-refining process.* J. G. P. Evans. U.S.P. (A) 1,368,642 and (s) 1,366,643, 25.1.21. Appl., (A) 11.11.19 and (s) 15.12.19.

(A) ONE end of a body of the oil is heated so that the temperature decreases towards the opposite end. The vapour arising from the hottest portion of the oil is brought into contact with the vapour arising from the cooler portions, whereby portions of the hotter vapour are condensed, the fractions condensed over successively cooler portions of the oil being collected separately. (s) A body of the oil in an upright conical vessel is heated to a higher temperature at the top than at the bottom. The vapour passes downwards around the walls of the vessel to effect a heat exchange between the vapour and the liquid, and the portions of liquid condensing from the vapour at different vertical heights are collected separately.—L. A. C.

*Gas; Apparatus for the manufacture of —.* J. E. Christopher, Assr. to T. H. Byrom. U.S.P. 1,367,512, 1.2.21. Appl., 28.2.20.

SEE E.P. 142,163 of 1918; J., 1920, 441 A.

*Paraffin wax emulsions; Manufacture of —.*  
G. W. Miles. E.P. 145,602, 29.6.20. Conv., 4.2.16.

SEE U.S.P. 1,168,534 of 1916; J., 1916, 299.

*Coke ovens, retorts and the like; Doors for —.*  
Ofenhau-Ges. E.P. 149,267, 13.7.20. Conv., 15.7.19.

See also pages (A) 173, *Ammonium sulphate* (U.S.P. 1,366,111); *Ammonium chloride* (U.S.P. 1,367,082). 179 *Hydrogen* (G.P. 328,691). 185, *Blast-furnace gas* (G.P. 326,485). 187, *Drying coke* (G.P. 328,529); *Siccatives etc.* (G.P. 327,374–5). 188, *Materials resembling rubber and linocyn* (G.P. 327,913).

**IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.**

*Yield of low-temperature tar from lignite.* Fischer and others. See III.

**PATENTS.**

*Coal, oil shale, wood, peat, and the like; Process and apparatus for the low and medium temperature carbonisation of —.* J. R. Garrow. E.P. 158,002, 31.7.18.

A ROTARY retort, resembling a cement kiln, in combination with a gas producer is used to effect the carbonisation of a fuel at low or medium temperature by the heat of the producer gas, without burning the latter. The direct contact possible between the hot gas and fuel gives a high thermal efficiency and, owing to the avoidance of local overheating, an improved yield of oil. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 13,830 of 1899, 18,279 of 1900, and 1676 of 1914; J., 1900, 725; 1902, 33; 1915, 604.)—C. I.

*Glow discharge lamps; Electric gas filled —.* A. Lederer. E.P. 12,153, 16.5.14. Conv., 19.5.13.

A GLOW discharge lamp of the type filled with one or more of the rare gases is rendered suitable for operation by an alternating current by arranging the electric circuit within the lamp to act as a rectifying valve of the Wehnelt type. Thus, one of the electrodes may consist of a refractory metal or conducting material covered with a metal compound or salt and so arranged that on starting the necessary heat is imparted to the covering to enable the lamp to light up. Alternatively, one of the electrodes may be considerably larger than the other.—J. S. G. T.



*Gas and vapour lamp; Electric* — J. Pintsch  
A.-G. G.P. 328,455, 28.10.15.

CADMIUM free from oxide and adsorbed gases, prepared by distillation in a high vacuum, is employed as cathode for the production of an arc discharge in a space containing a small quantity of one of the rare gases. In part of the discharge the cadmium vapour is made luminous, so that when neon is employed in the lamp, a white light results.  
—J. S. G. T.

*Metallic body to be enclosed in an evacuated vessel.*  
G.P. 330,372. See I.

### III.—TAR AND TAR PRODUCTS.

*Low-temperature tar from lignite; Variations in the yield of* — F. Fischer, W. Schneider, and A. Schellenberg. Brennstoff-Chem., 1921, 2, 52–58.

THE effect of drying lignite on the yield of low-temperature tar has been investigated. Taking the yield from freshly mined lignite as 100, the yield of tar from air-dried lignite was 74, from lignite dried at 105° C. in a stream of carbon dioxide 72, and from lignite dried at 105° C. in air 56. These differences are probably due to oxidation of the lignite by the oxygen in the air. The individual constituents of the tar do not diminish to an equal extent.—W. P.

*Benzol acid sludge; Treatment of* — Stephenson. Gas World, Feb. 5, 1921, Coking Sect., 19–20.

A TALL narrow still is unsuitable for the recovery of benzol from acid sludge, as frothing occurs. Steam should be introduced through a number of orifices. A still has been designed by the author on these lines; it also provides for a jet of ammonia gas being introduced into the swan-neck to neutralise the sulphur dioxide given off. Benzol suitable for the market is thus produced direct. 20% of benzol was recovered and the residual acid liquor varied in strength from 33 to 55%, calculated as H<sub>2</sub>SO<sub>4</sub>. The black resinous residue containing 1% ash is suitable as a fuel; or if washed with water, dried, and distilled, it yields 15% of oil. Up to 8% of pyridine was found in the acid liquor.—C. I.

*Aniline; Determination of — and its volumetric diazotisation.* T. Sabalitschka and H. Schrader. Z. angew. Chem., 1921, 34, 45.

ANILINE solutions, containing at least 2–3 mols. of sulphuric acid per mol. of aniline, and kept cold by means of ice-water, may be accurately titrated with N/1 sodium nitrite solution, with the aid of spot tests on potassium iodide-starch paper. The nitrite solution may be added several drops at a time at first, but as the reaction becomes slower the additions should be made drop by drop and the liquid must fail to react with the test paper before the next drop is added. The titration is continued until a positive reaction is obtained  $\frac{1}{4}$  hr. after the addition of the last drop. In order to reduce the error due to the withdrawal of numerous drops for the spot tests, an approximate preliminary titration is desirable. A titration occupies  $\frac{1}{2}$ –1 $\frac{1}{2}$  hr. The nitrate solution may be standardised against pure aniline or sodium sulphanilate. In test solutions containing 1.018 g. and 1.686 g. of aniline per 100 c.c., the authors found 1.015 g. and 1.689 g. respectively.—J. H. L.

*Cresols; Nitrobenzoates of the three* — E. Hänggi. Helv. Chim. Acta, 1921, 4, 23–25.

THE *o*-nitrobenzoyl derivatives of *o*-, *m*-, and *p*-cresol have the respective melting points, 68°–69°, 58.5°–60°, and 78°–79.5° C.; the *m*-nitrobenzoyl-

derivatives, 96.5°–97.5°, 63°–64°, and 77°–78° C., and the *p*-nitrobenzoyl-derivatives, 92°–93°, 85°–85.5°, and 96.5°–97.5° C. (Cf. J.C.S., Mar.)  
—T. H. P.

*Orthodichlorobenzol solvent.* Gardner. See XIII.

#### PATENTS.

*Transformer oil; Process for the manufacture of* — M. Melamid. E.P. 143,193, 14.2.20. Conv., 15.5.19.

AN anthracene oil fraction free from phenol is treated with oxidising agents, e.g., a mixture of potassium bichromate or permanganate and sulphuric acid, or the like, below 0° C., e.g., at –15° to –20° C., or below normal temperature, e.g., at 0°–5° C., in the presence of a suitable solvent, e.g., benzene. The supernatant oil is then separated from the resinous deposit, and is distilled *in vacuo*.  
—L. A. C.

*Sulphur compounds of the thiophene series; Manufacture of* — H. Scheibler. G.P. 327,050, 29.4.14.

THE crude tar oil from bituminous rock is heated with soda-lime, and after separation from the inorganic bases, the product is treated with alkyl magnesium halides. The oil obtained by decomposition of the organo-magnesium compounds is distilled over sodium, yielding a colourless or yellowish oil consisting of a solution of thiophene compounds in hydrocarbons. (Cf. J., 1920, 55a.)—L. A. C.

*p-Nitrotoluene-o-sulphonic acid; Manufacture of* — W. Osakeyhtö. G.P. 327,051, 9.7.18.

THE product obtained by sulphonating *p*-cymene is treated at 40°–60° C. with nitric acid, nitric acid and sulphuric acid, or with nitrates, and the *p*-nitrotoluene *o*-sulphonic acid is isolated as the sodium salt.—L. A. C.

*Diphenylene sulphide; Manufacture of* — Manufacture of diphenylene disulphide [thianthrene]. O. Lange, M. W. Widmann, and A. Wennerberg. G.P. (A) 330,833, 12.2.20, and (B) 330,834, 26.3.20.

(A) 2,2'-DIHYDROXYDIPHENYL, prepared by fusing diphenylene oxide from coal-tar with alkalis, is heated with phosphorus pentasulphide until evolution of hydrogen sulphide has almost ceased. The product on crystallisation from acetic acid or alcohol has m.p. 98.5° C., and gives a colourless solution in concentrated sulphuric acid. (B) A solution of catechol in mono- or dichlorobenzene is heated with phosphorus pentasulphide. The product forms colourless needles, m.p. 156° C., from acetic acid, and shows a characteristic violet colour with concentrated sulphuric acid.—L. A. C.

*Chloronaphthalenes.* G.P. 327,704. See XIII.

*Material resembling linxyn.* G.P. 327,913. See XIV.

### IV.—COLOURING MATTERS AND DYES.

*Java indigo plant; Conditions affecting the quality of the* — (Leaf yield and richness of the leaf in indigotin). W. A. Davis. Agric. Res. Inst., Pusa, Indigo Pubn. No. 7, 1–33.

WHEN indigo is grown with wheat as a cover crop, the indigo which follows the wheat grows slowly at first, probably owing to the removal of moisture from the soil by the wheat crop, but after the monsoon breaks it grows rapidly and forms a plant of exceptionally high quality containing a very high percentage of indigotin in the leaf. When the readily available nitrogen in the soil is high, the indigotin content of the plant is low, and although most nitrogenous manures give high yields of plant

per acre, the yield of dyestuff is low because of the low content of indigotin in the leaf. A cover crop improves the quality of the indigo by removing readily available nitrogen compounds from the soil. When the soil is poor in nitrogen, the plant is forced to utilise nitrogen fixed from the air by the nodular bacteria, and the activity of these organisms determines high indigotin content, the production of indican being due to the plant removing from the nodules nitrogen compounds which would be prejudicial to their continuous action. Favourable development of the nodules, which ensures a good growth of plant with a high indigotin content, is dependent on a low nitrogen content in the soil, a good supply of organic matter supplying carbohydrates, and a good supply of soluble phosphate in the soil. The very high yields obtained at Bunhar with mustard and linseed as cover crops indicate that, with proper manuring, indigo can be grown with a cover crop with great advantage, the poor results obtained hitherto in most cases being attributable to the depleted state of most plantation soils as regards phosphate and humus.

—F. M. R.

**6-Methylisatin.** J. Bonnefoy and J. Martinet. *Comptes rend.*, 1921, 172, 220—221.

*m*-TOLUIDINE condenses with mesoxalic esters to give esters of 6-methyl-oxindole-3-carboxylic acid, which on saponification in presence of air give an alkali isatate, from which on acidifying the free isatin is obtained. Carbon bisulphide reacts with *m*-toluidine in the presence of hydrogen peroxide to give di-*m*-tolylthiourea, which with potassium cyanide and lead carbonate in aqueous alcohol yields hydrocyanodi-*m*-tolylcarbonyl-imide,  $\text{CH}_3\text{C}_6\text{H}_4\text{NH}(\text{C}(\text{N})\text{N}:\text{C}(\text{N})\text{C}_6\text{H}_4)_2$ , which when heated at 40° C. in benzene with aluminium chloride for 4 hrs. gives 6-methylisatin-*m*-toluide. On boiling this with dilute hydrochloric acid 6-methylisatin is obtained, which when treated in acetic acid solution with the technical melt of phenylglycine gives methyl-6-indole-3-indole-2-indigo, m.p. 307°, dyeing wool and silk, and having a slight affinity for cotton.—W. G.

**Isatin-5-sulphonic acid.** J. Martinet and O. Dornier. *Comptes rend.*, 1921, 172, 330—331.

WHEN isatin is dissolved in fuming sulphuric acid containing 20%  $\text{SO}_3$ , and the mixture is heated on a water bath for  $\frac{1}{2}$  hr., isatin-5-sulphonic acid is obtained, identical in every respect with that formed by the oxidation of sodium indigo-5,5'-disulphonate. The phenylhydrazone of the acid furnishes a lemon-yellow potassium salt, which dyes wool and silk a yellowish green. Potassium isatin-sulphonate condenses with indoxyl in acetic acid solution to give indirubin, which in aqueous solution in an acid bath dyes wool and silk a violet shade.—W. G.

**Nitro-derivatives of carbazine, phenoxazine, and thiodiphenylamine; Absorption spectra of certain** — F. Kehrman and H. Goldstein. *Helv. Chim. Acta*, 1921, 4, 26—31.

THE authors have investigated the absorption spectra of a number of nitro-compounds in alcoholic sodium hydroxide solution, in which they exhibit an intense characteristic coloration clearly differentiated from that of the solid compounds. This property is shown only by compounds possessing at least one nitro group in the para-position to the cyclic nitrogen atom. (*Cf.* J.C.S., Mar.)

—T. H. P.

**Colouring matters; Determination of the constitution of — from their absorption spectra.** F. Kehrman and M. Sandoz. *Helv. Chim. Acta*, 1921, 4, 31—44. (*Cf.* J., 1920, 224A.)

THE authors have examined the absorption spectra

of monoamino-derivatives of methylphenazine, aposafranine, and its isomerides and acetyl-compound, and of their mono-, di-, and tri-acid salts. From comparison of the spectra of the different compounds deductions are drawn as to the constitutions. (*Cf.* J.C.S., Mar.)—T. H. P.

**Furfural.** Monroe. *See* XX.

**Phthaleins.** Csángi. *See* XXIII.

#### PATENTS.

**Phthaleins; Manufacture of —.** O. Imray. From Monsanto Chemical Works. E.P. 157,030, 31.5.20.

AN anhydrous aromatic sulphonic acid, with or without zinc chloride, is used as the condensing agent. Thus, 100 pts. of phthalic anhydride is mixed with 200 pts. of phenol, solution is effected at 80° C., 200—250 pts. of, say, toluenesulphonic acid, or a mixture of 175 pts. of toluenesulphonic acid and 50 pts. of anhydrous zinc chloride, is added, and the mass is kept at a temperature below 140° C., preferably about 120° C., for 10—12 hrs. The product, which is particularly free from tarry by-products and from ortho-condensation products, is washed free from phenol and acid with water, and is finally purified by precipitation from sodium hydroxide solution in the usual way. The yield approximates to the theoretical.—G. F. M.

**Monoarylaminoquinones; Manufacture of sulphonic acids of —.** M. Becke and W. and H. Suida. G.P. 326,511, 23.5.18. Addn. to 300,706 (J., 1920, 399 A).

THE desired compounds are produced by the interaction of 2 mols. of a quinone and 1 mol. of an aminoarylsulphonic acid in alcoholic solution. An aqueous solution of sodium sulphanilate (1 mol.) is added to an alcoholic solution of 1,4-benzoquinone (2 mols.) with stirring; after standing, the sodium salt of *p*-sulphophenylaminobenzoquinone separates as copper-coloured needles, which are readily soluble in water, slightly soluble in alcohol, and insoluble in ether, and dye wool an orange-brown shade from an acid bath. The monoarylaminoquinones serve as dyestuffs and for the production of mixed diaryldiaminoquinones, either as such or on the fibre.—L. A. C.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Wool; Action of water on —.** A. Reychler. *Bull. Soc. Chim. Belg.*, 1920, 29, 291—300. *Chem. Zentr.*, 1920, 92, II., 139.

ON heating wool to temperatures up to 150° C. with water or alkaline or acid solutions, under normal or increased pressure, acid or basic substances pass into solution which are precipitated by Fuchsin and form lakes with Eosin or Ponceau. The undissolved wool has lost the property of double refraction between crossed nicols, and can be powdered when dry. If wool is heated to 110°—120° C. with water, its elasticity is diminished, but it is still very tenacious, and its plastic properties at these temperatures are utilised in finishing processes. The relative insensitiveness to acids of methyl orange dyed on wool or silk, which is even more marked at high temperatures, is due to the combination of the indicator with a system which, although not markedly basic, is capable of combining with acid molecules.—L. A. C.

**Cellulose in bast fibres.** Y. Uyeda. *J. Ind. Eng. Chem.*, 1921, 13, 141—143.

RENKER's modification of Cross and Bevan's method (direct chlorination without preliminary treatment

with alkali) is recommended for the determination of cellulose in bast fibres. The function of pectin substances in bast fibres is discussed, and it is suggested that the cellulose of the hemp fibre has, in some degree, an oxycellulose structure.—W. P. S.

*Cellulose mucilage* J. E. Minor. J. Ind. Eng. Chem., 1921, 13, 131–133.

THE first stage in the decomposition of cellulose is the formation of mucilaginous soluble dextrins which are at once adsorbed by pure cellulose, yielding an aggregate termed hydrocellulose; mucilage differs from hydrocellulose in the larger proportion of soluble adsorbed dextrins present. The adsorbed dextrins act as catalysts in the hydrolysis of cellulose and complete hydrolysis leaves only soluble dextrins. Immersion in cold water produces slow hydrolysis of cellulose; hot water hastens hydrolysis and weakens adsorption, so that mucilaginous products are destroyed as rapidly as they are formed. Pulps with an original high copper value can be converted into mucilage by beating more easily than those with low copper values, owing to the larger amount of catalyst (dextrins) present; the copper value of bleached pulp can be increased by careful acid treatment previous to beating. Owing to its more colloidal state, cellulose mucilage decomposes a salt and adsorbs the metal ion more readily than does pure cellulose. The action of moisture or acid on cellulose aids dyeing by forming mucilage.—W. P. S.

*Kraft [paper] pulps; Testing — for strength.* K. G. Wilen. Paper, Dec. 20, 1920, 15–17, 36.

KRAFT pulps (sulphate pulps) are used at present not only in the manufacture of pure "Kraft" paper, but also in the making of various other papers and boards, and it is important to have a standard method for testing the pulp, especially when mixtures of pulp are used. The following method has been used by the author for three years:—25 g. of air-dried pulp is thoroughly broken up by forcing the pulp through a screen by means of a jet of water. The screened pulp is placed in a tared weighing cylinder, made up to 3 kg. with water, and transferred to a ball mill jar. The pulp is beaten for one hour in a ball mill; the beaten pulp is washed out, made up to 10 l., and three portions of 450 c.c. each are placed in a mill and made up to approximately 5 l. with water. After thoroughly stirring, the contents are poured on to a sheet machine. The sheets are pressed for a few seconds under hand pressure, dried at 180° F. (82° C.), and allowed to re-absorb moisture for 1 hr. Pieces 6"×6" are then cut, and weighed to the nearest 0.05 g. The weight of the sheets is calculated to lb. per ream of 480 sheets, 24"×36", i.e., the commercial weight standard for "Kraft." The sheets are then tested on a Mullen tester. The average Mullen test multiplied by 100, and divided by the ream weight of the sheets, is the expression for the strength of the pulp.—J. C. K.

*Wilkinite, a new loading material [for book paper].* S. D. Wells. Paper, Dec. 8, 1920, 19–21, 34.

WILKINITE, or "jelly rock," a highly colloidal clay, can be used as a substitute for English china clay as a filler for paper. The best results were obtained by using a mixture of the two minerals, viz., in the proportion of 90% of wilkinite and 10% of china clay. Wilkinite will remain in suspension for a long period, and has the power of holding in suspension English china clay up to at least twice the amount of wilkinite present. When a mixture of the two fillers is used, the greater the proportion of wilkinite, the greater the retention of filler by the paper.—J. C. K.

*Maize cobs.* La Forge. See XIX A.

#### PATENTS.

*Silk; Process for degumming —.* O. Goldschmidt. E.P. 131,906, 28.8.19. Conv., 4.12.16.

SILK is degummed by subjecting it to the conjoint action of a peroxide or per-salt and soap solution. 60 g. of raw material is treated with a cold 0.1% solution of sodium peroxide, boiled, without removing the peroxide, in a 0.2% solution of soap for 5–10 mins., rinsed, and dried. Alternatively, the material is moistened with 20 kg. of 10% hydrogen peroxide solution and then boiled for 10–15 mins. in a solution containing 12 kg. of soap in 1400 kg. of water. The process is specially suitable for low-grade qualities of silk and the product dyes more readily and is more glossy than that obtained by the usual methods.—A. J. H.

*Alkali-cellulose; Manufacture of matured — and its instantaneous conversion into matured xanthate [viscose].* A. Thilmann. G.P. 323,035, 15.3.19.

CELLULOSE or the like is ground *in vacuo* at a raised temperature with an alkaline solution of about 15° B. (sp. gr. 1.116) until the alkali has increased by evaporation to a suitable strength, when the necessary quantity of carbon bisulphide is added to form a sufficiently matured xanthate.—L. A. C.

*Cellulose solutions for spinning artificial threads or the like; Process of preparing durable cupric ammonia —.* Glanzfäden A.-G. E.P. 145,035, 14.6.20. Conv., 12.10.17.

SEE G.P. 306,107 of 1917; J., 1918, 575 A.

*Wool washing effluent.* E.P. 157,490. See I.

*Washing and cleansing agents.* G.P. 327,635. See XII.

#### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Dyeing; Co-ordination theory of valency in relation to adjective —.* G. T. Morgan. J. Soc. Dyers and Col., 1921, 37, 43–47.

ACCORDING to Werner's co-ordination theory, the chemical affinity of an atom is divisible in a great variety of ways, depending on the circumstances of chemical combination, into a variable number of valencies of unequal intensities, which are distributed uniformly over the spherical surface of the atom. The chemical elements do not all give rise to equally stable co-ordination complexes, and in regard to this difference the volume of the atomic sphere plays an important part. The metals which give rise to stable co-ordination complexes are those which yield the lakes produced in adjective dyeing with acid mordant colours. Certain compounds and compound radicals, termed chelate groups, take up two positions round the central atom and function as adjective dyes on suitably mordanted material. The lakes or chelate complexes of a number of dyes are reviewed, and it is concluded that the co-ordination theory of valency suffices not only to explain and correlate known facts, but will serve as a guiding principle in future investigations on the theory and practice of adjective dyeing.—F. M. R.

*Indigo vat; Influence of the degree of dispersion of the dyestuff in the — on the shade produced.* M. Freiburger. Textilber., 1921, 2, 84–86.

THE colour of a fabric dyed with indigo is due to indigo deposited within the fibre, this being greyish-blue in colour and very fast to rubbing; to a cellulose-dyestuff compound of indigo-blue colour, fast to rubbing; to crystalline indigo deposited near the surface of the fibre, violet-blue in colour and less

fast to soap and rubbing; and to re-oxidised indigo attached to the surface of the fibre, bluish-grey in colour and not fast to soap and rubbing. The degree of dispersion of the indigo in the vat determines the condition in which it dyes the fibre. Fast dyeings result when the indigo is in a highly dispersed state, and this is obtained by increasing the temperature and dilution of the dye liquor. Fugitive dyeings result when the dye liquor contains indigo in a highly aggregated state, due, e.g., to the presence of an excess of lime or glue or an insufficiency of caustic soda. The sodium salt of indigo white forms a colloidal solution of a higher degree of dispersion and enters the fibre more freely than the calcium salt, and it also serves as a carrier of re-oxidised indigo.—A. J. H.

*Staple fibre fabrics; Dyeing and finishing of* — A. Winter. *Textilber.*, 1921, 2, 40—41.

STAPLE-FIBRE fabrics (cf. J., 1919, 281 A) containing vegetable fibres only are prepared for dyeing by the usual methods except that bleaching is preferably carried out by immersing the fabric for 20—30 mins. at 20° C. in a 1½% solution of sodium hypochlorite, lightly rinsing, immersing in hydrochloric acid of 1° B. (sp. gr. 1.007), washing, and treating with an "antichlor." Fabrics containing animal fibres are bleached with hydrogen peroxide, sodium peroxide, or sodium perborate solutions, rinsed, and soured in formic acid. Dyeing may be carried out in the usual manner with direct, sulphur, and vat dyestuffs. Fabrics used for clothing generally contain wool and are dyed by the usual methods for cotton-wool unions, very fast shades being obtained by dyeing the wool with chrome-mordant dyes and the cotton with sulphur dyes, glue, glucose, or bisulphite being added to the dye-bath to preserve the wool from injury. Shot effects may be obtained. After dyeing the wool is invariably treated with formic acid to restore its pleasing "handle." The finishing processes are dependent on the composition of the fabric, but are similar to those usually adopted. 5—6% of Naphthylamine Black EFF (Cassella) in the presence of 4—5% of sulphuric or formic acid gives a full shade on wool and a light grey on staple-fibre, so that it is very useful in testing staple-fibre fabrics for the presence of animal fibres.—A. J. H.

*Silk; Dyeing* — with indanthrene colours. H. E. Schroers. *Textilber.*, 1921, 2, 38—40.

For \*Anthraflavon G.C.; Indanthrene Scarlet G.S. pdr.; Indanthrene Gold Orange R and R.R.T.; \*Indanthrene Violet R extra, RR extra, RT and B extra; Indanthrene Blue G.C. and GCD; Indanthrene Blue-green B; Indanthrene Red-brown R; \*Indanthrene Green B; Indanthrene Grey B and 3B; \*Indanthrene Olive G. pdr.; and \*Indanthrene Brown B conc., dyeing is carried out with 10 kg. of material for 1 hr. at 40° C. in a dye-bath containing 250 l. of liquor, 3 l. of caustic soda of 40° B. (sp. gr. 1.386), 500—1500 g. of hydrosulphite pdr. conc., 1—40% of dyestuff, 1200 g. of glycerin, and 5000 g. of "cell-pitch" 50%. For \*Anthraflavon GC; \*Indanthrene Gold Orange G; Indanthrene Orange RT; Indanthrene Copper R; Indanthrene Red-brown R; Indanthrene Red G. and R; \*Indanthrene Ross B; Indanthrene Bordeaux B and B extra; Indanthrene Violet R extra, RR extra, and RT; Indanthrene Blue GCD; \*Indanthrene Green B; Indanthrene Grey B and 3B; \*Indanthrene Olive G pdr., and \*Indanthrene Brown B conc., the alkali content of the dye-bath is reduced to 750 c.c. of caustic soda of 40° B and the amount of glycerin is increased to 1250 g. During dyeing the temperature of the bath is raised to 40° C. Dyestuffs marked \* are used in a cold or lukewarm dye-bath. After dyeing the material is left to oxidise in the air (drying should be avoided), allowed to hang

over-night, well washed in water, treated in a boiling solution of soft soap, and brightened with formic acid. A firmer "handle" is obtained if the silk is treated before soaping in a 0.6% solution of tannic acid, and fixed with tartar emetic. Silk having a soft "handle" and suitable for embroidery is obtained by treating the material, after brightening, in a neutral bath containing a 0.3% solution of Marseilles soap and some olive oil emulsion. In dyeing with gold orange dyes, oxidation with air is supplemented by working the dyed silk for 30 mins. in an acid bath containing 750 g. of sodium perborate and 1 l. of 30% acetic acid (or its equivalent of formic acid) per 250 l. of liquor, the temperature being increased from 40° to 70° C. The shades produced with the above-named dyes are very fast to washing, acids, bleeding, hydrogen peroxide, and light except in the following cases:—Indanthrene Olive G pdr. and Brown B conc. are only moderately fast to hydrogen peroxide; Indanthrene Orange RT, Copper R, Rose B, and Olive G pdr. are not fast to light; and Indanthrene Gold Orange R and RT, Violet RT and B extra, and Red-brown R are only moderately fast to light. In certain cases useful shades may be obtained by treating the dyed material with an acid and sodium nitrite.—A. J. H.

*Detergents [for fabrics]; Determination of the washing and cleaning action of* — P. Heermann. *Textilber.*, 1921, 2, 37—38, 61—62.

FABRIC is artificially soiled by immersion in a 0.5% solution of 20% colloidal indigo paste, pressed until it contains its own weight of liquor, and dried at the ordinary temperature. It may be further similarly treated with starch paste and a solution of machine oil in benzene or ether. Samples of the soiled fabric (10 g.) are cleansed by squeezing 20 times at room temperature in 250 c.c. of water, of 0.4% solution of sodium carbonate, of 0.4% solution of soap powder, or of a solution containing 0.125% of palm oil soap (60% fat) and 0.075% of sodium carbonate. The liquor, contained in a porcelain dish, is heated on the water-bath during 10—15 mins. to 50° C. and the fabric again squeezed 20 times. The dish is then removed from the water-bath, and after each of three intervals of 15 mins. the fabric is squeezed 20 times (100 times in all), washed in lukewarm water till no more colour is extracted, washed in cold running water, dried, and pressed with a hot iron (sublimation of the indigo must be avoided). Washing must always be carried out with distilled water. The liquids mentioned have an increasing cleansing power in the order given, and for the last this is considered to be 100. Samples of the artificially soiled fabric are cleansed, in the same manner, in solutions of the detergent under examination and then compared with the standards. The degree to which a fabric has been cleansed is judged from its colour, and not from its loss in weight. The results obtained by this method agree with those obtained in large-scale practice.—A. J. H.

#### PATENTS.

*Discharge printing.* C. H. Boehringer Sohn. G.P. 328,817, 9.10.17.

THE addition of salts of hile acids, e.g., cholic acid, increases the solubility of the discharge products and facilitates their removal from the fibre.

—L. A. C.

*Textile goods; Apparatus for the treatment of* — in hank form by means of circulating liquids. E. Nägelin. U.S.P. 1,367,494, 1.2.21. Appl., 26.4.20.

THE apparatus contains a device by means of which liquid is supplied under slight pressure to a number of horizontal hank carriers. The carriers are tubes perforated in their upper side only and mounted

in a bearing at one end, so that they can be rotated; the liquid flows from the perforations down along the hanks or skeins.—A. J. H.

*Fabrics; Production of coloured ornamental designs upon textile — by machine embroidery and the application of liquid dyes or colours.* Straub and Co. E.P. 157,682, 10.6.20. Conv., 1.3.20.

A STENCIL of paper, celluloid, or other material is pressed against the fabric, preferably while in the frame of the embroidery machine, either before, during, or after the embroidery, and is sprayed with a viscous solution of the dyestuff.—A. J. H.

*Cleansing [fabrics etc.] with alkali salts of protalbinic and lysalbinic acids; Method of —.* C. Bennert, Assr. to The Chemical Foundation, Inc. U.S.P. 1,367,007, 1.2.21. Appl., 27.12.15.

SEE G.P. 311,542 of 1915; J., 1919, 625 A.

*Dyeing or similarly treating yarns in the form of hanks, skeins, or bundles; Machine for —.* A. Ashworth. U.S.P. 1,367,112, 1.2.21. Appl., 14.4.20.

SEE E.P. 137,980 of 1919; J., 1920, 361 A.

*Bleaching, dyeing, or the like; [Wire] foundations for supporting sliver, roving, yarn, or the like during the process of —.* H. Winslow. E.P. 157,507, 20.9.19.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Sulphuric acid manufacture; Use of ammonia oxidation units for —.* W. Wyld. Chem. Age, 1921, 4, 150—151.

IN connexion with the employment of ammonia oxidation units for sulphuric acid manufacture, some details of plant design are given. Loss of ammonia from storage tanks is prevented by connecting the vent with a water-scrubber. Access to the platinum gauges is afforded by providing a removable cover with mica window in the converter. To economise platinum, two layers of wire of 0.0125 in. diam. and 120- to 150-mesh may be used instead of four layers of 0.005 in. diameter and 80-mesh. Special precautions must be taken in fixing the asbestos washers when introducing the catalyst, to prevent disintegration of the asbestos, and lead piping should be used, instead of iron, from the still head to converter, to avoid deposition of rust on the catalyst. The efficiency of the oxidation units is easily maintained at 90%, and the production is 1.5 tons of nitric acid per sq. ft. of catalyst per 24 hrs.—W. J. W.

*Sulphur dioxide and sulphur trioxide; Determination of — in burner gases by means of the gas absorption flask.* H. Krull. Papierfahr., 1921, 19, 93—95.

THE author gives results of his analyses of sulphur gases by means of a modified Reich gas absorption flask (Chem.-Zeit., 1920, 43, 167). The estimation is more rapidly made than with the Orsat apparatus, and comparative results showed an average difference of 2.2%.—W. J. W.

*Hydrochloric acid; Utilisation of —.* H. Hackl. Chem.-Zeit., 1921, 45, 149—150.

IT is suggested that weak hydrochloric acid be employed for the treatment of crude phosphate, instead of the usual sulphuric acid, dicalcium phosphate being then precipitated by means of milk of lime. This would permit of the sulphuric acid from the cheaper native pyrites being used for the production of the hydrochloric acid and sodium sulphate instead of for superphosphate manufacture as at present.—A. de W.

*Ammonia; Microchemical identification of gaseous — as hexamethylenetetramine picrate.* C. Kollo and V. Teodossiu. Bul. Soc. Chim. România, 1920, 2, 100—102.

WHEN a drop of a 40% solution of formaldehyde saturated first with picric acid and then with hexamethylenetetramine picrate is exposed on a microscope slide in an atmosphere containing a trace of ammonia, characteristic microscopic crystals of hexamethylenetetramine picrate are deposited.

—W. G.

*Potassium compounds; Measurement of the vapour pressure of certain —.* D. D. Jackson and J. J. Morgan. J. Ind. Eng. Chem., 1921, 13, 110—118.

As determined by the Von Wartenberg method (Z. anorg. Chem., 1912, 79, 76; Z. Elektrochem., 1913, 19, 482) the vapour pressures of potassium compounds were found to be as follows, the results being given in mm. of mercury:—Hydroxide, 8 at 795° C.; chloride, 1.54 at 801° C., 8.33 at 948° C., 24.1 at 1044° C.; oxide in carbonate, 1.88 at 970° C., 5.0 at 1130° C.; sulphate, 0.4 at 1130° C.; natural silicates, 0 at 1335° C. From the results obtained with potassium chloride together with the b. pt. of this compound as given by Bergstrom (Med. Finska Kemistsamf., 1915, 24, 2), the Nernst vapour pressure formula for potassium chloride has been calculated to be:  $\log P = -5328/T + 1.75 \log T + 0.000511T - 0.7064$ .—W. P. S.

*Iodides; Determination of — in the presence of iodates.* V. Thüringer. Bul. Soc. Chim. România, 1920, 2, 73—77.

FOR the quantitative analysis of a mixture of iodides and iodates, the iodate is first estimated by adding 0.2 g. of the substance dissolved in water to a solution of 1.5 g. of potassium iodide acidified with sulphuric acid and titrating the liberated iodine with N/10 sodium thiosulphate. To another 0.2 g. of the substance dissolved in water a known volume of N/60 iodic acid is added, the mixture is acidified with sulphuric acid and boiled until the whole of the liberated iodine has disappeared. The solution is cooled, an excess of potassium iodide is added, and the iodine liberated titrated with N/10 sodium thiosulphate. From these two titrations the iodide present in the original mixture can be calculated.—W. G.

*Uranium; Preparation of pure compounds of —.* E. Wiike-Dürfurt. Wiss. Veröffentl. Siemens-Konzern, 1920, 1, 143—146. Chem. Zentr., 1921, 92, I, 170—171.

THE uranium oxide obtained from cleveite by solution of the mineral in nitric acid, followed by removal of the rare earths and thorium by oxalic acid, and of the heavy metals by hydrogen sulphide and ammonium sulphide in the usual way, was found to contain small amounts of alkali, probably as uranate. This was removed by precipitation of the uranium as disulphide instead of as uranyl sulphide. The process was repeated and the sulphide dissolved in hydrochloric acid, the solution treated with ammonia, and the precipitate dissolved in nitric acid. The oxide obtained from the nitrate crystallised from this solution showed no trace of alkali when examined in the spectroscope. Recrystallisation of uranyl nitrate from ether does not give a product free from alkali.—A. R. P.

*Topochemical reactions. Formation of carbon at contact substances.* V. Kohlschütter and A. Nageli. Helv. Chim. Acta, 1921, 4, 45—76. (Cf. J., 1919, 174 A, 221 A.)

THE structure of the carbon deposited when carbon monoxide is passed through a tube containing a heated metal is influenced by the character of the

contact metal, the temperature of the reaction, and the state of subdivision of the metal. Cobalt yields a graphitic carbon of markedly different nature from that obtained with iron or nickel; the similarity of the results obtained with electrolytic and powdered cobalt indicates a specific action of the metal. Neither with reduced nor with powdered iron is the formation of graphite favoured by raising the temperature of the reaction; increase in the degree of subdivision of the metal does, however, favour the development of the graphitic type. In comparison with the carbon of electrolytic deposits, which exhibits strongly graphitic properties, that deposited on metallic powders, with the exception of powdered cobalt, more nearly resembles soot in appearance and behaviour. The view that a continuous gradation exists between the different forms of carbon is thus supported, and it is further evident that, under similar external conditions, the formation of one or another product may be obtained from one and the same chemical reaction merely by alteration of the structure of the locus of the reaction.—T. H. P.

Carbon; Fusion of —. E. Ryschkewitsch. Z. Elektrochem., 1921, 27, 57–61.

A RESISTANCE furnace is described consisting essentially of a carbon box, 10 cm. square, with open ends 7 cm. square, into which fit the carbon electrodes. The inside of the box is filled with graphite. A current of 500–560 amps. was passed for 8–12 hrs. At the end of this time the electrode ends and the contents of the box were found to be pure graphite, at least 99.9%, and the electrode ends showed, when examined microscopically, unmistakable signs of having been in the molten condition. At ordinary pressures the temperature range over which carbon can remain in the fluid condition is very restricted, and this accounts for the difficulty of obtaining evidence of melting.

—E. H. R.

Point discharge in nitrogen. Pirani and Lax. See XI.

Nitric acid. Winkler. See XXIII.

#### PATENTS.

Sulphuric acid; Manufacture of —. J. H. Brown. U.S.P. 1,365,964, 18.1.21. Appl., 16.7.18.

In a sulphuric acid chamber plant means are provided for washing the burner gases, and for reheating the washed gases previous to oxidation.

—W. J. W.

Sulphur trioxide; Making — [by the contact process] and apparatus therefor. H. M. Weber, Assr. to The Ellis-Foster Co. U.S.P. 1,366,439, 25.1.21. Appl., 8.2.17.

THE relative speed of the gases in the inner and outer sections of the catalytic members is adjusted by varying the cross-section, porosity, and depth of the members.—C. I.

Hydrochloric acid free from arsenic; Production of —. H. Frischer. G.P. 326,618, 21.2.19.

HYDROCHLORIC acid gas containing arsenic compounds is treated with hydriodic acid gas or iodides whereby insoluble arsenic tri-iodide is formed.—A. R. P.

Copper sulphate; Manufacture of —. R. E. Bea. E.P. 147,689, 8.7.20. Conv., 3.8.17.

In a vessel containing dilute sulphuric acid are suspended a perforated receptacle containing copper waste into which dips a leaden anode, and a porous pot filled with dilute sulphuric acid in which hangs the leaden cathode. Deposition of copper on the latter is avoided by this arrangement, and copper sulphate crystallises in the vessel.—W. J. W.

Thorium compounds [double metaphosphate and sulphate of thorium]; Manufacture of insoluble —. H. Wade. From Lindsay Light Co. E.P. 156,892, 11.10.19.

A DOUBLE metaphosphate and sulphate of thorium,  $\text{Th}(\text{PO}_3)_2\text{SO}_4$ , of the same composition, but purer than the compound obtained by heating a natural mineral containing thorium and phosphates, such as monazite sand, with sulphuric acid at a high temperature (cf. U.S.P. 1,323,735; J., 1920, 517 A) is prepared in the form of white acicular crystals by dissolving 120 g. of thorium sulphate,  $\text{Th}(\text{SO}_4)_2 \cdot 8\text{H}_2\text{O}$ , in 50 c.c. of 80% phosphoric acid and heating for 10 hrs. at  $280^\circ\text{C}$ . Water and sulphuric acid fumes are evolved and the product forms a nearly solid crystalline mass, insoluble in water or in dilute acids. The same substance is also obtained by heating thorium phosphate with an equal weight of sulphuric acid, preferably in presence of 20–40% of phosphoric acid, for 10 hrs. above  $260^\circ\text{C}$ .

—G. F. M.

Thorium nitrate; Method of manufacturing —. H. N. McCoy, Assr. to Lindsay Light Co. U.S.P. 1,366,123, 18.1.21. Appl., 30.12.18.

As a step in the manufacture of thorium nitrate, monazite sand is treated with concentrated sulphuric acid, and the insoluble thorium compound produced, after removal of soluble impurities, is converted into thorium hydroxide.—W. J. W.

Lead sulphate; Process of making — direct from lead sulphide ores. P. A. Mackay. E.P. 157,554, 18.10.19.

FINELY-GROUND galena, substantially free from zinc, is added gradually, with constant stirring, to an excess of sulphuric acid containing about 20% of free sulphur trioxide, whereby a vigorous exothermic reaction takes place and substantially pure lead sulphate is produced. The excess of acid is recovered by diluting the mass with water, allowing the precipitate to settle, and decanting the clear liquid. The lead sulphate is suitable for use as a pigment.—A. R. P.

Ammonium sulphate; Apparatus and process for the recovery of —. J. Becker, Assr. to The Koppers Co. U.S.P. 1,366,111, 18.1.21. Appl., 18.6.17.

MOIST gases, freed from tar, are passed through a saturation bath in which ammonia is absorbed, and are then superheated and passed through another part of the bath, thus promoting evaporation and precipitation of ammonium sulphate.—W. J. W.

Ammonium chloride; Manufacture of solid — from hydrochloric acid gas and ammonia. A.-G. für Anilin-Fabr. G.P. 326,619, 14.2.9.

HYDROCHLORIC acid gas and ammonia are allowed to react in the presence of a fine spray of water in such quantity that it is practically completely vaporised by the heat of the reaction, which is thereby enabled to go on to completion.—A. R. P.

Ammonium chloride; Method of producing — from fuels. W. Ostwald, Assr. to The Chemical Foundation, Inc. U.S.P. 1,367,082, 1.2.21. Appl., 23.2.17.

THE nitrogen in fuels is obtained as ammonium chloride by injecting into the burning fuel an aqueous solution from which chlorine is evolved.

—C. I.

Ammonia; Process for recovering liquid — from moist ammonia vapours. "Metan" Spółka z Ograniczoną Odp. we Lwowie. G.P. 326,929, 13.6.18. Conv., 8.8.17.

THE vapours are cooled by passing them through a column and then through a cooler, in each of which



an increase of pressure, somewhat greater than the vapour pressure of the ammonia at the temperature of the cooler, is produced, preferably by passing into the apparatus highly compressed water-vapour.—A. R. P.

*Metallic halides; Process of making* —. C. M. Alexander. U.S.P. 1,366,626, 25.1.21. Appl., 9.10.19.

A PULVERISED metal-bearing material, fuel, air, and a halogen are injected together into a combustion and reaction chamber.—C. I.

*Ferrous oxide; Process of producing* —. A. J. MacDougall, Assr. to H. C. Bigbird, J. G. Rowe, and R. H. Bennett. U.S.P. 1,367,409, 1.2.21. Appl., 21.8.20.

IRON is treated in an electrolytic cell with the acid product of the electrolysis of a salt solution, and the ferrous salt thus formed is decomposed by the alkaline product of electrolysis.—C. I.

*Potassium nitrate and mixed salts containing potassium and nitrogen; Manufacture of* —. Badische Anilin und Soda Fabr. G.P. 306,334, 11.3.16.

AMMONIUM nitrate and potassium chloride are dissolved in hot water; potassium nitrate crystallises on cooling, and a mixed salt, suitable for use as a fertiliser, is separated from the mother liquor either by evaporation to dryness or by fractional crystallisation. If the potassium chloride employed contains much sodium chloride, the quantity of water is so adjusted that the greater part of the sodium chloride remains undissolved in the hot solution.—L. A. C.

*Magnesium; Manufacture of a double salt for use in the electrolytic production of* —. Aluminium- und Magnesium-Fahr. A.-G. G.P. 323,413, 3.12.15.

THE mixture of salts obtained as by-product in the electrolytic production of magnesium from carnallite is mixed with the end liquor from the production of potassium chloride, the solution is partially evaporated, and is allowed to crystallise. The double salt  $MgCl_2 \cdot KCl \cdot 6H_2O$  crystallises out together with sodium chloride, and the water of crystallisation is eliminated by heat. The fused salt,  $MgCl_2 \cdot KCl \cdot NaCl$  is allowed to cool in a vessel of inverted conical shape, whereby impurities collect in the apex.—L. A. C.

*Hydrogen sulphide; Production of* — from sulphates. Apparate-Vertriebs-Ges.m.b.H. G.P. 306,352, 20.11.17. Addn. to 304,231 (J., 1920, 364 A).

As a reducing agent for sulphates, carbon monoxide, alone or mixed with hydrogen and hydrocarbons, may be employed instead of gaseous hydrocarbons.—W. J. W.

*Calcium or barium sulphide; Manufacture of* — from calcium or barium sulphate. Metallbank und Metallurgische Ges. A.-G. G.P. 307,612, 3.3.17.

A MIXTURE of the sulphate with coal or the like is made into small lumps before heating in a rotary tube furnace.—L. A. C.

*Nitrogen compounds; Production of* —. "Azot". Ges.m.b.H. G.P. 309,154, 4.5.18.

ATMOSPHERIC air, or other mixture of oxygen and nitrogen, mixed with steam, is forced as a blast over or against strongly heated metals, such as

platinum, nickel, or iron, or other heat-resisting bodies. Ammonium nitrite may thus be produced by blowing a mixture of air and steam over heated platinum gauze.—W. J. W.

*Alkali peroxides; Preparation of dry, hydrated* —. Drägerwerk H. u. B. Dräger. G.P. 310,671, 6.12.16.

HYDRATION of alkali peroxides is effected in a liquid medium which is immiscible with water and unaffected by the reacting materials (water and alkali peroxide), and the liquid is afterwards separated. Petroleum, ethylene chloride, and the like may be employed.—W. J. W.

*Manganese acetate; Production of* — from higher oxides of manganese. Konsortium für Elektrochem. Ind. G.m.b.H. G.P. 327,086, 30.3.19.

A MIXTURE of acetic acid and acetaldehyde is allowed to react with the higher oxides of manganese. Addition of an alkali or alkaline-earth acetate accelerates the reaction.—A. R. P.

*Potassium ferricyanide; Manufacture of* — from calcium potassium ferrocyanide. C. Giesen. G.P. 327,289, 18.1.20.

CALCIUM potassium ferrocyanide is suspended in water and, either before or after oxidation with chlorine, the requisite quantity of potassium chloride is added to replace the calcium. Potassium ferricyanide crystallises on evaporating the solution and is thus easily separated from the calcium chloride.—L. A. C.

*Calcium chloride; Manufacture of fused* —. A. Riedel. G.P. 327,867, 16.5.17.

HYDROCHLORIC acid gas, or waste gas containing it, is led through a tower packed with lumps of calcium carbonate. The calcium chloride solution formed is concentrated by the heat of reaction as it passes down the tower, and the liquor which collects at the bottom solidifies on cooling.—L. A. C.

*Hydrogen; Production of* — by alternate oxidation and reduction of iron. W. Paternoster. G.P. 323,691, 8.3.18.

IN a hydrogen generator in which the contact material is arranged in layers, steam and other gases are introduced for oxidation and reduction purposes respectively in the form of divided streams, the combined cross-sections of which are greater than that of the part of the apparatus through which they pass.—W. J. W.

*Hydrogen peroxide; Processes for the production of* —. L'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude. E.P. 141,758, 17.4.20. Conv., 11.5.14.

SEE F.P. 476,816 of 1914; J., 1916, 634.

*Aluminium nitride; Production of* — by electrically generated heat. Armour Fertilizer Works. Assecs. of M. Shoeld. E.P. 146,919, 27.10.19. Conv., 10.7.19.

SEE U.S.P. 1,344,153 of 1920; J., 1920, 570 A.

*Peroxide of nitrogen; Process for the manufacture of* —. L. Duparc and C. Urfer. U.S.P. 1,366,773, 25.1.21. Appl., 24.9.19.

SEE E.P. 133,041 of 1919; J., 1921, 147 A.

SEE also pages (A) 169, *Pump for acids* (E.P. 145,803 and 146,409). 170, *Acid receptacle* (U.S.P. 1,366,170). 183, *Roasting furnace* (G.P. 326,441). 197, *Potassium chlorate* (G.P. 300,714).



## VIII.—GLASS; CERAMICS.

*Screens for optical pyrometry.* Forsythe. See XXIII.

## PATENTS.

*Glass.* M. A. Smith, Assr. to McKee Glass Co. U.S.P. 1,365,797, 18.1.21. Appl., 15.7.18.

A GLASS having a low coefficient of expansion and high resistance to changes of temperature is made from a batch consisting of a preponderance of silica, smaller amounts of boric oxide, alumina, sodium nitrate, and sodium carbonate, and still smaller amounts of calcium carbonate and arsenic trioxide.—H. S. H.

*White clouded glasses, enamels, and glazes; Process for manufacture of —.* E. Rietz. U.S.P. 1,366,101, 18.1.21. Appl., 22.8.19.

IN the production of white clouded glass, enamels, and glazes, unpurified clouding material is used, together with sufficient material containing fluorine to prevent discoloration.—H. S. H.

*Silica; Method and apparatus for washing and treating —.* R. H. Moore. U.S.P. 1,366,582, 25.1.21. Appl., 29.8.17.

SILICA is passed through a classifier consisting of a series of tanks each with a sorting compartment, provided with an overflow, stirring mechanism, and an endless conveyor placed at an angle, with its lower end in the compartment and its upper end above one side of the stirring mechanism in the next compartment. Means are provided for washing the coarse sand as it is conveyed from one compartment to the next.—A. B. S.

*Cement suitable for dental purposes; Preparation of a —.* C. Jacobsen. G.P. 329,005, 24.10.19.

THE product is obtained by precipitation from a solution of suitable materials dissolved in the form of their salts etc. For example, the product obtained by precipitation with ammonia of a solution of alum, zinc sulphate, phosphoric acid, and water-glass is washed, dried, calcined, powdered, and sifted. It is three times denser than the corresponding mixture of powdered aluminium silicate, zinc silicate, zinc phosphate, and alumina, and the cement is tougher.—G. F. M.

*Glass-furnace.* A. F. Peeters, Assr. to Naaml. Vennoten. Glasfabriek Leerdam. U.S.P. 1,366,745, 25.1.21. Appl., 4.4.18.

SEE E.P. 123,146 of 1918; J., 1919, 222 A.

*Clay and the like; Mining of —.* J. S. Highfield. U.S.P. 1,366,466, 25.1.21. Appl., 7.11.19.

SEE E.P. 135,277 of 1918; J., 1920, 65 A.

## IX.—BUILDING MATERIALS.

*Concrete; Effect of tannic acid on the strength of —.* D. A. Abrams. Proc. Amer. Soc. for Testing Materials, 1920, 20, Pt. I. Bull. 7, Structural Materials Res. Lab., Lewis Inst., Chicago.

ABOUT 2000 tests were made on samples of concrete prepared from aggregates containing up to 0.4% of tannic acid, the latter being applied as a surface coating and regarded as being generally typical of the organic impurities existing in natural sands. The mixes varied from 1:5 to 1:2, the size of aggregate from fine sand to  $\frac{1}{4}$ -inch graded material, and the age of the test-pieces (stored in damp sand) from 7 days to 2 years. For all percentages of tannic acid in the aggregate the strength of the concrete was reduced for all mixes and ages. In some cases the reduction amounted to 50% when the aggregates contained 0.1% of tannic acid, but the weakening effect of the latter was less marked as the percentage increased beyond this figure.

Lean mixtures were more affected by tannic acid than rich ones, and those containing the finer were less affected than those containing the coarser aggregates. The reduction in strength of the concrete was found to be a function of the concentration of tannic acid in the mixing water; the results indicated that wetter mixes would be less affected than drier ones by the presence of organic impurities. The strength of the 7- and 28-day specimens was reduced to a greater extent by tannic acid than that of the 1- and 2-year specimens; some of the 1:5 mixes, containing the finer sands and the higher percentages of tannic acid, disintegrated before the time of test. The results are regarded as indicating the fundamental dependence of the strength of concrete on the quantity of mixing water as expressed by the water-ratio.—W. E. F. P.

## PATENTS.

*Wood; Process for drying —.* Holzbearbeitungsges. m.b.H., K. Haring, and E. Voit. E.P. 141,727, 14.4.20. Conv., 28.3.19.

THE wood is exposed uniformly on all sides to a source of heat radiation and at the shortest possible distance from it, the temperature being preferably low at first, e.g., 30°–40° C., and then raised by stages to about 90° C. It is possible to interrupt the operation after drying at the lower temperatures and to proceed later with the expulsion of the water from the inner layers at the higher temperatures. It is advantageous to conduct the drying operations in a vacuum.—H. S. H.

*Wood; Method and apparatus for transforming [and colouring] —.* H. F. Weiss, Assr. to C. F. Burgess Laboratories. U.S.P. 1,366,225, 13.1.21. Appl., 6.10.17.

WOOD is heated to 830°–420° F. (194°–215° C.) in circulating air for a sufficient time to transform it into a uniformly coloured, less hygroscopic product.—A. B. S.

*Cement and by-products; Process of making —.* S. B. Newberry. U.S.P. 1,366,479, 25.1.21. Appl., 1.6.20.

CEMENT clinker is made by heating a finely ground mixture of raw cement materials and carbonaceous fuel. The condensable products volatilised from the fuel are collected and recovered, and the heating of the clinker is effected by gas volatilised from the fuel and by the combustion of the fixed carbon.—A. B. S.

*Kiln; Portland cement and calcining —.* Cement kiln. J. Nelson. U.S.P. (A) 1,366,585 and (B) 1,366,586, 25.1.21. Appl., 19.7.19 and 24.3.20.

(A) A SHAFT-KILN with ring gas-burners fixed internally is covered by a hood through which the kiln can be fed by a conveyor. Across the bottom of the hood is a screen and a stirring and distributing blade rotated by an electric motor, so that the charge is uniformly distributed over the contents of the kiln. (B) In a similar kiln having spaced annular walls, a downdraught chamber communicates with the lower part of the space between the annular walls, this space being fitted with baffle plates so that it forms a spiral updraught flue having its outlet near the top of the kiln.—A. B. S.

*Cement; Manufacture of porous —.* A. Grosche. G.P. 327,907, 9.5.19.

A SUITABLE powdered metal, e.g., 1% of zinc dust, is added to the dry cement, which is then mixed with a solution of a compound, e.g., a 2% solution of calcium chloride or sodium hydroxide, which reacts with the metal, at the temperature generated by the heat of reaction, to liberate gas bubbles and thus render the cement porous.—L. A. C.

*Artificial stone; Manufacture of* — M. E. Lehlanc and K. Gerson. G.P. 327,906, 13.2.14.

A MIXTURE of bauxite, or residues from the manufacture of aluminium, and 10–15% of slaked fat lime or hydraulic lime, with or without the addition of quartz sand, is worked up according to the usual method for lime-sand bricks, yielding stone showing a high resistance to pressure and atmospheric influences such as frost.—L. A. C.

## **X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.**

*Sulphur; Estimation of* — in wrought iron, steel, and cast iron. A. Cartoceti. Giorn. Chim. Ind. Appl., 1920, 2, 682–686.

THE following method is recommended as a check on the more rapid methods used in industrial laboratories for estimating sulphur in iron or steel. 10 g. of the sample, in filings or turnings, is mixed with 2 g. of potassium chlorate and the mixture treated in a porcelain dish with 100 c.c. of concentrated nitric acid containing 2 c.c. of bromine; the acid is added slowly and intense heating of the liquid avoided by placing the dish in cold water. When evolution of gas ceases, 20 c.c. of concentrated hydrochloric acid is added and the solution evaporated to dryness on a sand-bath. The sand is then removed from the bath and the dish heated on the metal tray to redness to decompose nitrates and complete the oxidation. The cold residue is again evaporated with 50 c.c. of concentrated hydrochloric acid, the residue being then evaporated with another 50 c.c. of the acid to a syrup, which is diluted with a little water and allowed to cool. The cold liquid is poured, with continual shaking, into a 1-litre measuring flask containing 300 c.c. of 10% ammonia solution, the dish being also rinsed into the flask. When cold the solution is made up to volume, mixed, the voluminous precipitate allowed to settle, and the liquid filtered through a dry filter. 500 c.c. of the filtrate is evaporated to about 150 c.c. and precipitated at the boiling point with 20 c.c. of boiling 10% barium chloride solution, which is added all at once and with stirring. It is necessary to carry out a blank estimation with the reagents.—T. H. P.

*Chromium in steels; Determination of small quantities of* — B. S. Evans. Analyst, 1921, 46, 38–42.

FOUR g. of the sample is dissolved by heating with 30 c.c. of sulphuric acid (1:3) and 20 c.c. of water, 10 c.c. of nitric acid is added, and the solution boiled to expel nitrous fumes; 25 g. of ammonium phosphate and 250 c.c. of water are then added, the boiling solution is treated with a slight excess of permanganate, boiled for a further 15 mins., and then poured into 120 c.c. of 20% sodium hydroxide solution which has been heated to boiling, treated with an excess of permanganate and then with 10 c.c. of 5% manganese sulphate solution. The resulting mixture, if acid in reaction, is rendered strongly alkaline with sodium hydroxide, cooled, acidified with acetic acid, diluted to 500 c.c., and filtered. In the absence of nickel and cobalt, the chromium is determined directly, by comparing the colour of the filtrate with that of a standard dichromate solution in the presence of sulphuric acid; if nickel and cobalt are present, 100 c.c. of the filtrate is boiled, treated with sodium hydroxide until a precipitate separates, cooled, filtered, the filtrate acidified with sulphuric acid, and the chromium then determined colorimetrically as described.—W. P. S.

*Molybdenum steel; Manufacture of* — Pokorny. Z. Metallkunde, 1920, 12, 238–239. Chem. Zentr., 1921, 92, II., 114–115.

ON account of the readiness with which molybdenum oxidises and the easy solubility of the oxide in the solid metal, the best way of adding molybdenum to steel is as a ferro-molybdenum alloy containing about 45% Mo, which has been melted electrically and carefully deoxidised. The best deoxidisers for such steels are titanium and vanadium, as they act also as nitrogen scavengers, their nitrides being readily volatilised, whereas when aluminium and silicon are used, difficultly volatile nitrides of molybdenum are formed. The double iron-molybdenum carbide is very sparingly soluble in molten iron; it is, therefore, advisable in preparing molybdenum steels to add to a bath low in carbon the required amount of a low-carbon ferro-molybdenum and then to carburise the metal up to the required carbon content.

—A. R. P.

*Copper; Metallographic investigation of electrolytically deposited bright* — G. Grube and V. Reusz. Z. Elektrochem., 1921, 27, 45–52.

SMOOTH, mirror-like deposits of copper were obtained by the method originally described by Müller and Bahntje (J., 1906, 484). The electrolyte consisted of a 0.5 molar solution of copper sulphate, acidified with sulphuric acid to give a *N*/100 solution of  $H_2SO_4$ , with the addition of 10 c.c. of a 1% gelatin solution to 200 c.c. of the copper sulphate solution. The copper electrodes were each 6.5 by 6.0 cm. and the current density was 0.035 amp. per sq. cm. During the first hour of the electrolysis the deposit remained bright; after this it became matt, but by adding a further 1 c.c. of the 1% gelatin solution every hour, it was possible to maintain the brilliancy of the surface of the deposited copper. The polished surface, etched with nitric acid and examined under high magnification ( $\times 400$ ), showed a high degree of homogeneity distinct from the crystalline character of ordinary electrolytic copper. A section perpendicular to the surface showed alternate layers of copper and gelatin deposited in a regular manner, indicating two alternate stages in the deposition process. The thickness of each layer was about 0.0056 mm. and the time required for the completion of each double layer would average 8.2 mins. The first stage in the deposition process probably consists in the formation of a film of a disperse copper-gelatin phase in which the gelatin, acting as a dispersion medium, retains the discharged copper for some time in the disperse phase. The second stage consists in the precipitation of the copper in a coarser form with consequent formation of a copper and a gelatin layer. The process is then repeated. The hypothesis receives support from measurements which were made of the cathode potential, which showed a periodic variation corresponding roughly with the time, calculated from the microscopical examination of the section, required for the completion of the two stages.—E. H. R.

*Copper; Metallographic investigation of electrolytically deposited bright* — G. Grube. Z. Elektrochem., 1921, 27, 53–54.

AN acknowledgment and short discussion of the work of Sieverts and Wippelmann (J., 1916, 604). The different results obtained by these workers from the results recorded in the previous abstract, were probably due to the much stronger gelatin solutions used by Grube and Reusz. Instead of parallel layers of gelatin and copper, Sieverts and Wippelmann observed a fine network structure in sections perpendicular to the plane of deposition.

—E. H. R.

*Electrolytic zinc plant of the Anaconda Copper Mining Co., at Great Falls, Mont.* F. Laist, F. F. Frick, J. O. Elton, and R. B. Caples. *Chem. and Met. Eng.*, 1921, 24, 245—250.

A detailed description is given of the plant and process used in the recovery of zinc by electrolysis from the complex zinc ores of Butte, containing about 33% Zn and 20% Fe. The ore is roasted in mechanically rabbled multiple-hearth furnaces of the Wedge type at temperatures not exceeding 600°—625° C., in order to produce the maximum amount of zinc sulphate. The flue dust is collected in a Cottrell plant and returned hot to the first hearth, as it has been found that the sulphur trioxide it contains increases the yield of zinc sulphate in the calcine. The roasted ore is leached in two stages; in the first, the whole charge is treated with about half the necessary sulphuric acid partly in the form of spent electrolyte containing 11.5% H<sub>2</sub>SO<sub>4</sub> and 7.5% Zn, and partly in the form of a slightly acid liquor obtained from the second stage of the leach, which is carried out with fresh acid, the resulting filtered solution being partly purified before adding to the fresh charge of calcine. The neutral solution obtained in the first stage, containing about 75% of the total soluble zinc, is treated with a small quantity of limestone and  $\frac{1}{4}$  of the total weight of calcine, whereby part of the copper and all the gelatinous silica and ferric iron are precipitated and carry down the arsenic and antimony. The clarified solution is treated successively with zinc sludge and zinc dust to remove in two stages the copper and cadmium, which are subsequently recovered from the slimes. The purified zinc sulphate solution is electrolysed in lead-lined wooden tanks containing 27 rolled sheet aluminium cathodes and 28 pure lead anodes, the distance between successive cathodes being 4 in. and between anode and cathode 1 $\frac{1}{2}$  in. The best deposits are obtained with a current density of 22—25 amps. per sq. ft. and by the addition every 2 hrs. of 1—1.5 oz. of glue per ton of metal deposited. The cathodes are removed when 40 lb. of metal has been deposited, stripped, and the zinc melted under ammonium chloride. The residue from the leaching of the roasted ore is smelted in reverberatories to produce a leady matte containing most of the copper, gold, and silver, a fume containing the greater part of the lead, and a slag with about 11% Zn, which can be worked up later.—A. R. P.

*Zinc; Metallurgy of* —. Lemarchands. *Rev. Mét.*, 1920, 17, 803—841.

In continuation of previous work (J., 1920, 338 A) the author has investigated the reactions between zinc oxide and carbon, zinc oxide and carbon monoxide, and zinc and carbon monoxide and finds that zinc oxide is reduced both by carbon and carbon monoxide, the former reaction starting at about 600° C. depending on the rate of heating, and the latter between 350° and 400° C., while the reverse reaction between carbon monoxide and zinc takes place at very high temperatures and is even then very incomplete. The results obtained are discussed from the point of view of the phase rule and the earlier work on the subject is reviewed and criticised.—A. R. P.

*Aluminium alloys; Heat treatment of* —. W. Fraenkel and R. Seng. *Z. Metallkunde*, 1920, 12, 225—237. *Chem. Zentr.*, 1920, 92, II., 115—116.

The authors have studied the heat treatment of aluminium alloys containing 1.5% Cu, 3% Cu, 4% and 8% Zn respectively, together with the effect of additions of 0.1% and 0.5% Mg. For the determination of the lowest temperature to which the alloy must be heated before quenching for improvement in the mechanical properties to take place and the highest temperature at which the effects of heat

treatment will persist, the test pieces were subjected to the impact test, as the Brinell hardness number did not give satisfactory results. Alloys free from magnesium showed no noticeable improvement on heat treatment; the copper alloys required a higher temperature than those containing zinc and the resulting metal could be heated to a higher temperature than the zinc alloys without deterioration. The temperature required rises with the content of alloying metal, while the temperature at which the effects disappear depends to a considerable extent on the rate at which the metal has been heated and the time that has elapsed since the heat treatment. The density and specific heat of the metal are not altered appreciably by heat treatment, but the electrical resistance rises immediately the lowest temperature is reached at which improvement takes place, and as the temperature increases further the rate of increase of resistance falls off until eventually a constant value is attained. When the temperature rises to such a point that the effects disappear, the resistance of the alloy reverts to its original value. These facts are consistent with a reaction in a homogeneous system during heat treatment. The authors attribute the effects of heat treatment to the formation from the homogeneous mixed crystal phase of a chemical compound which is soluble in the mixed crystals. The alloy after heat treatment is in the metastable condition.—A. R. P.

*Copper-aluminium alloy; Mechanism of solidification of a* —. J. D. Edwards. *Chem. and Met. Eng.*, 1921, 24, 217—220.

The densities of various copper-aluminium alloys containing up to 60% Cu were determined at 20° C. for the solid metal and at temperatures between the melting point and 1000° C. for the liquid metal. On plotting the values found at 20° C. and also their reciprocals (i.e., the specific volumes of the alloys) against the copper content of the series, it was found that the latter curve was a straight line and hence the specific volume and, therefore, the density of any copper-aluminium alloy containing 0—60% Cu could be calculated for any temperature up to its melting point from the known densities and coefficients of expansion of the constituent metals. The mechanism of solidification of No. 12 alloy (commercial aluminium with 8% Cu) was studied by calculating and plotting the densities of the solid and liquid phases in equilibrium during the process of solidification of the alloy and by the construction of an inverse rate cooling curve. The alloy commences to freeze at 636° C. and is completely solid at 540° C., while at 577° C. there is a point of inflexion in the cooling curve due to the separation of the aluminium-silicon eutectic. The density of the first crystals formed (at 636° C.) is greater than that of the liquid from which they separate; they therefore tend to sink, but at 630° C. the densities become equal and at lower temperatures the crystals float in the liquid. The phenomena are discussed in relation to the cooling of an ingot of the alloy.—A. R. P.

*Gold-copper alloys; Anomaly in the expansion of* —. A. Portevin and J. Durand. *Comptes rend.*, 1921, 172, 325—327.

The very great brittleness of copper-gold alloys containing 20% or more of copper disappears on tempering owing to a transformation. This transformation is accompanied, on heating, by a sudden expansion. With an alloy containing 25.2% of copper this anomaly occurs at 400° C. during heating and at 350° C. on cooling. The anomaly becomes less important when the alloy has been previously heated to a temperature equal to or exceeding that at which the transformation during heating occurs.—W. G.

**Molybdenum; Determination of** — J. P. Bonardi and E. P. Barrett. U.S. Bureau of Mines, Techn. Paper 230, 1920. 33 pp.

THE following procedures are recommended for the determination of molybdenum in ores, especially in low-grade ores and tailings from the flotation process. **Volumetric method.**—0.5–5.0 g. of ore, containing not more than 0.08 g. Mo, is dissolved in *aqua regia* and the solution evaporated with 20 c.c. of sulphuric acid (1:1). After dilution, the solution is neutralised with ammonia, treated with 10 c.c. excess, boiled, and filtered. The precipitate is washed with hot water, re-dissolved in sulphuric acid, and re-precipitated as before. The combined filtrates are neutralised with sulphuric acid, 12.5 c.c. added in excess, the solution heated to 70° C. and passed through a Jones reductor 20–30 in. long, filled with amalgamated zinc. The reduced solution, which contains the molybdenum as sesquioxide, and should be of a bright green colour, is received in 50 c.c. of a solution containing 50 g. of ferric ammonium sulphate, 45 g. of manganese sulphate, 87.5 c.c. of syrupy phosphoric acid, and 87.5 c.c. of strong sulphuric acid per litre, and the resulting colourless solution titrated with *N*/20 permanganate (1 c.c.=0.0016 g. Mo). The ore may also be decomposed by fusion with six times its weight of sodium peroxide in an iron crucible, in which case the solution of the melt in water is made up to 500 c.c., 250 c.c. filtered through a dry paper, neutralised with sulphuric acid, 12.5 c.c. excess added, and the solution boiled, reduced, and titrated as before. If the ore contains copper, arsenic, or antimony the acid solution before being passed through the reductor is treated with a few c.c. of copper sulphate solution and several grms. of zinc, when part of the arsenic and antimony is evolved with the hydrogen and the remainder precipitated with the copper. The filtered solution is reduced as before. When tungsten or vanadium is present, as is usually the case with wulfenite ores, the molybdenum is precipitated by hydrogen sulphide under pressure from the solution after just acidifying with sulphuric acid and adding 2 g. of tartaric acid. The sulphide is dissolved in *aqua regia* and the solution evaporated with sulphuric acid as before. **Gravimetric method.**—The ore is dissolved in *aqua regia* as described above and the nitric acid eliminated by evaporation with hydrochloric acid, or with 10 c.c. of sulphuric acid if lead is present. The diluted solution is filtered and the filtrate boiled with an excess of ammonia, the precipitate being re-dissolved and re-precipitated with ammonia. Before filtering the solution it is treated with magnesia mixture if phosphorus is present. The combined filtrates are acidified with hydrochloric acid, 5 c.c. excess added, followed by 5–10 g. of sodium acetate, and the liquid titrated hot with lead acetate solution (18 g. per litre) until a test shows no colour with tannic acid. 2 c.c. excess of lead acetate is added and the liquid allowed to stand hot for 15–20 mins., then filtered, and the precipitate washed with 2% ammonium acetate or nitrate solution, dried, ignited together with the paper, and weighed as  $PbMoO_4$ . In this method lime and sulphur do not interfere, tungsten and vanadium are removed as in the volumetric method, arsenic is removed with magnesia mixture, and copper remains in the filtrate from the lead molybdate.

—A. R. P.

**Formation of carbon at contact substances.** Kohl-schütter and Nägeli. See VII.

#### PATENTS.

**Magnetic alloy sheets; Method of controlling grain growth in** — V. B. Browne. U.S.P. 1,367,015, 1.2.21. Appl., 6.6.19.

**SILICON steel ingots** are gradually heated to about 1200° F. (650° C.), then raised to forging tempera-

ture, rolled into sheet form, and finally annealed. During rolling the necessary heating between passes is carried out in an oxidising atmosphere and the pressure exerted upon the sheets in the final pass or passes is controlled in accordance with the temperature, whereby a predetermined grain size is produced in the finished sheets after annealing.

—J. W. D.

**Alloy steels; Process of producing** — C. M. Johnson. U.S.P. 1,366,662, 25.1.21. Appl., 12.12.19.

IN smelting iron oxide both ferrosilicon and ferrochromium are introduced into the charge of the smelting furnace.—J. W. D.

**Electrodes, welding rods, and soldering sticks.** E. H. Jones, and Alloy Welding Processes, Ltd. E.P. 157,534, 14.10.19.

MINERAL graphite, retort graphite, manufactured graphite, or a mixture thereof is incorporated with other alloying materials in electrodes, welding rods, and soldering sticks used for soldering etc. by the electric arc or blowpipe, more particularly such as are required to provide a certain amount of carbon in the deposited metal. The quantity of graphite employed varies from 3% for nickel steel to 4% for cast iron.—J. S. G. T.

**Vanadium; Method of recovering** — from its ores. P. A. Mackay E.P. 157,555, 18.10.19.

ONE part of the vanadium ore, e.g., lead vanadate, crushed to 200-mesh, is added gradually with constant stirring to 1.5 parts of fuming sulphuric acid. When the vigorous exothermic reaction has subsided a reducing agent, e.g., sulphur dioxide or hydrochloric acid, is added to the mass to convert the vanadium into vanadyl sulphate. This may also be brought about by addition of a carbonaceous material to the mixture during the reaction. The mass is treated with water, the insoluble lead sulphate and gangue are removed by filtration, and the vanadium is recovered from the solution by electrolysis, or by precipitation with alkalis.—A. R. P.

**Furnaces; Heat treatment** — [for metals]. W. A. Russell, and The Manchester Furnaces, Ltd. E.P. 157,696, 25.8.20.

TWO fires at opposite sides of the furnace are supplied with only a limited quantity of air through the fire-bars, and additional secondary air is admitted to the products of combustion while they are in side passages leading from the fire-boxes to the working chamber; the secondary air is preheated by passing it through passages in the walls adjacent to the fire-boxes, and the waste gases are exhausted through ports in the back and mouth of the working chamber.—B. M. V.

**Roasting furnace for pyrites, spent oxide, or the like; Rotary** — Rocholl. G.P. 326,441, 22.10.18.

THE interior of the furnace is divided into a number of cells parallel to the long axis, and a central conveyor charges them through a series of radial channels near the front end of the cells, whilst the gases pass out through openings in front of the channels into a chamber surrounding the conveyor, whence they pass to the scrubbers etc. This method of construction allows the gases to be collected continuously during the charging of the furnace.—A. R. P.

**Roasting and sintering; Apparatus for** — Fellner and Ziegler. G.P. 327,007, 7.8.19.

THE material passes down a vertical shaft furnace, and at the bottom is fed through an automatically adjusted opening into a horizontal rotating tube furnace. The hot gases leaving the latter furnace,

in which the material is sintered, pass up the shaft furnace and dry and preheat the material therein.

—L. A. C.

*Coating iron, steel, and certain other metals with aluminium; Process for* — J. Thompson, Ltd., W. J. Thompson, and H. E. Partridge. E.P. 158,010, 25.7.19.

THE surfaces to be coated are first rubbed with pure linseed oil or other suitable oil, then heated to burn off the oil, after which one or more coatings of oil are applied while warm, and, finally, aluminium, or powdered aluminium and a flux, or a paste of oil, powdered aluminium, and a flux is dusted or sprayed on and the metallic articles heated to a suitable temperature to melt the aluminium and flux and burn off the oil. The process is repeated until the necessary thickness of aluminium is obtained, or sheet aluminium of desired thickness may be sweated on to the aluminium coating.—J. W. D.

*Sherardising drums.* S. O. Cowper-Coles. E.P. 158,154, 20.2.20.

A SHERARDISING drum is provided with a central longitudinal heating tube or with two or more similar tubes disposed eccentrically about the axis. By this means the hot gases for heating the apparatus may be passed through the tube or tubes and over the whole of the outside surface of the drum, or *vice-versa*, thus ensuring rapid and even heating of the contents. The apparatus is mounted on rollers to allow of rotation, and rests on a carriage running on rails so that it may be moved into and out of the furnace.—A. R. P.

*Alloys; Method of preparing metallic — and of refining metals and alloys.* H. C. Hall, and Rolls-Royce, Ltd. E.P. 158,156, 25.2.20.

ALLOYING metal or metals, before being introduced into the molten main constituent, are granulated and coated with oil either by melting them and pouring into a suitable oil or by pouring them on to or over a roller or series of rollers immersed in the oil.—J. W. D.

*Slag; Process of and apparatus for making* — J. Lund. U.S.P. 1,366,398, 25.1.21. Appl., 13.4.18.

MOLTEN slag is purified by charging it into a furnace and heating from below, whilst an oxidising gas is applied to the upper surface.—A. B. S.

*Flotation cell and method; Pneumatic —. Flotation separatory process and apparatus.* J. M. Callow, Assr. to Pneumatic Process Flotation Co. U.S.P. (a) 1,366,766 and (b) 1,366,767, 25.1.21. Appl., (a) 26.4.15, and (b) 27.4.14.

(A) In order to prevent the formation of a bed of impermeable pulp at the bottom of the separator cell in the pneumatic flotation process, the cell is divided into compartments by means of parallel walls reaching nearly to the bottom and, at these points of restricted communication between the compartments, eddy currents are produced so as to prevent the settling of any material. (s) An apparatus for separating the metalliferous constituents of an ore from the gangue consists of a pulp receptacle provided with means for forming uniformly distributed bubbles within the pulp and for exhausting them from the surface.—A. R. P.

*Suspensions [of ores and the like]; Method for working up* — A. Nathansohn. G.P. 325,706, 23.12.19. Addn. to 323,836 (J., 1921, 120 A).

SUSPENSIONS of ores containing substances sensitive to light, such as uranium minerals or chrome iron ore, are subjected without preliminary treatment to the action of light until the electrical charge on the

particles is modified. Addition of substances sensitive to light, as specified in the principal patent, is, in these cases, unnecessary.—A. R. P.

*Lead; Process for recovering — from ores etc.* S. Hultdt. G.P. 325,897, 9.2.18. Conv., 10.3.17.

THE charge is heated electrically to a temperature sufficient to melt it and the slag produced in the reaction. The greater part of the lead settles out on, and may be tapped from, the hearth, while that vaporised is recovered as metal in a condenser from which air is excluded.—A. R. P.

*Lead; Process for the removal of — from mixtures of metallic vapours. Process and apparatus for separating the vapours of metals or metallic compounds of greatly differing specific gravities, e.g., lead and zinc.* K. Recke. G.P. (A) 325,971, 21.10.17, and (b) 325,972, 23.10.17.

(A) METALLIC vapours containing lead are passed over precipitating agents which are decomposed by heat, e.g., sulphates, carbonates, oxides, or carbides or a mixture of these. The lead in the gases thereby combines with the volatile part of the precipitating agent, e.g., sulphur trioxide from sulphate, and the resulting lead compound is subsequently precipitated, leaving the vapours substantially free from lead. (B) The stream of vapours passing through a horizontal flue impinges on a vertical wall with an extension at the upper end sloping upwards, the height of the wall depending on the relative proportions of the two vapours present. The heavier vapour is, by this means, directed out of the flue into a separate condensing chamber while the lighter vapour flows over the top of the wall to its own condensing chamber. The sloping extension gives the heavier vapour an opportunity to condense or flow back along a longer distance, so that it is almost completely removed from the mixture.—A. R. P.

*Aluminium; Process for rendering — passive.* O. Pieschel. G.P. 325,943, 3.1.17.

ALUMINIUM, especially that to be used in accumulators, may be rendered passive to sulphuric acid by treatment with ammonia, followed by heating.—A. R. P.

*Aluminium; Process and apparatus for the purification of — containing less than 1% of impurities.* Physik.-Tech. Reichsanstalt. G.P. 326,160, 23.1.19.

THE metal is slowly heated to its melting point and the more easily fusible alloy containing the impurities is pressed out from the mass of pure aluminium crystals. The operation is carried out in an electrically heated cylindrical tube provided with a glass or earthenware funnel at the lower end standing on a small receptacle of similar material to collect the impurities. A fireclay rod closely fitting into the top of the cylinder acts as a pestle with which to press together the purified crystals. Aluminium containing 0.18% Fe and 0.22% Si can be purified by several repetitions of the process so that the final product contains 99.95% Al. (Cf. Mylius and Mylius, J., 1921, 150 A.)—A. R. P.

*Bauxite; Process of smelting — for production of ferro-aluminium in an electric furnace.* Rheinische Elektrowerke A.-G. G.P. 327,055, 16.11.18. Addn. to 308,542 (J., 1920, 754 A).

THE alumina in the slag is removed, with consequent increase in value of the slag as a substitute for carbide, by addition of iron or iron compounds and, if necessary, of coal to the slag, with subsequent separation of the metallic aluminium produced thereby.—L. A. C.

*Metals of high melting point, e.g., tungsten; Process for melting* — J. R. Gault. G.P. 326,424, 21.3.18.

THE metal is melted in a crucible constructed of the same metal and supported in a graphite crucible lined with an oxide of the metal. An opening in the bottom of the graphite crucible is closed by a piece of the metal under treatment which melts when the contents of the crucible are completely fused and allows the contents to flow out.

—L. A. C.

*Blast-furnace gas; Process for the dry purification of* — E. Raffloer. G.P. 326,485, 3.4.17.

THE gases are passed, first through a thin layer of a valuable filtering medium, e.g., fine ore, coke or coal breeze, in order to precipitate the heavier particles of metallic dust, then through a thicker layer of a less valuable filtering medium, e.g., ground slag, to precipitate the lighter and finer non-metallic particles. The former medium is changed more frequently than the latter and, before use, is mixed with a binding material, preferably of such a nature as to remove the harmful acids and moisture from the gases. Lime is most suitable for this, as it assists in the subsequent briquetting of the material for the recovery of the values therein.

—A. R. P.

*Metallic hydrosols; Production of electro-colloidal* — Chem. Fabr. von Heyden A.-G. G.P. 326,655, 16.2.19.

HYDROSOLS of metals which are oxidised by electrical disintegration under water are made by carrying out the process in the presence of a reducing agent, e.g., sodium hydrosulphite, catechol, pyrogallol, hydroxylamine, or hydrazine, and of a protective colloid. The reducing agent may be removed from the solution by dialysis or by precipitating the metallic colloid, washing, and re-dissolving it in the absence of air, as the purified colloid is readily oxidisable.—A. R. P.

*Agglomerates; Production of* —, for the blast furnace, from fine ore, flue-dust, burnt pyrites, and fuel. C. Giesecke. G.P. 327,248, 21.12.17.

FLUE-DUST, burnt pyrites, and finely divided ore and fuel are mixed with sufficient water and pressed into briquettes which are sintered in a shaft furnace, without drying. By firing the briquettes wet they do not break before sintering.—A. R. P.

*Steel; Stable surface alloy* — P. A. E. Armstrong. E.P. 143,829, 17.7.19. Conv., 24.5.19.

SEE U.S.P. 1,322,511 of 1919; J., 1920, 117 A.

*Electrode for use in depositing molten metal [for welding], and process of making same*. W. H. Boorne. E.P. 157,509, 30.9.19.

*Double magnesium salt*. G.P. 328,413. See VII.

## XL.—ELECTRO-CHEMISTRY.

*Electric furnaces; Operating details of* — E. T. Moore. Chem. and Met. Eng., 1921, 24, 171—176.

THE paper is based upon a questionnaire submitted to steel manufacturers operating electric furnaces in the United States. When the transformer primary voltage employed exceeds 22,000 volts, the electric furnace service is expensive to instal and operate. All surges should be kept within the closest limits possible. Systems of installation wherein the transformers are located directly behind the furnace, and tilting occurs away from the transformers, require somewhat longer flexible leads than

the system of installation in which the furnace tilts at right angles to the line from the transformer to the furnace. In the former system, however, there are no side strains on the electrode masts. Furnace conductors should be installed so as to avoid unnecessary eddy currents. With furnaces of up to one or two tons capacity little is gained by installing double voltage control. In larger furnaces, a high voltage should be used during part of the melting period, and a lower voltage during the refining period. The cost of electrodes amounts to about .9% of the total operating expense of a furnace, and that due to breakage of carbon electrodes amounts to a further 7.7% on the average. The average consumption of carbon electrodes amounts to 39.9 lb. per ton of metal produced. A specification of the lengths, tolerances, diameters, ends, threading, and resistivity of amorphous carbon and graphite electrodes is detailed. The average specific resistance per inch cube at 20° C. of carbon electrodes is not to exceed 0.002 ohm, and for no single electrode is it to exceed 0.0025 ohm. The average specific resistance of graphite electrodes is to be 0.00036 ohm per inch cube. A new type of water-jacketed ring for cooling the electrodes and the port-holes of the furnace roof is described. Amorphous carbon electrodes in which a hard bonding material is used have recently been made. Silica bricks are those chiefly used in electric furnace construction. Magnesite bricks are being increasingly used for the construction of parts of the furnace in contact with the charge and for parts exposed to extremely high temperatures. Dolomite has been successfully employed for the construction of furnace bottoms. The use of the electric furnace in the brass industry is rapidly increasing. The power consumption varies from 174 k.w. hrs. per ton of metal in the case of continuous operation of induction furnaces to 240—500 k.w. hrs. per ton for intermittent operation of arc and resistance types.—J. S. G. T.

*Silent electric discharge; Use of the* — for the detection of fire-damp and for gas analysis. G. Erlwein and H. Becker. Wiss. Veröffentl. Siemens Konzern, 1920, I, 71—75. Chem. Zentr., 1921, 92, II., 210.

VARIATIONS of current strength occur when methane is present in the air flowing through a Siemens ozone tube in which a constant potential difference is maintained. Such variations of current strength are obtained with gaseous mixtures in which chemical action can take place between the constituents. The presence of hydrogen or methane in air causes a considerable increase of current strength. The variations of current strength can be detected by means of a galvanometer of the thermo-electric type. By employing an intensifier tube in conjunction with a relay, signals can be operated indicating the presence of certain gases. The variations of current strength may also be used to determine the concentration of the gas admixed with the air.

—J. S. G. T.

*Point electrical discharge in nitrogen. [Determination of minute quantities of impurities.]* M. Pirani and E. Lax. Wiss. Veröffentl. Siemens Konzern, 1920, I, 167—178. Chem. Zentr., 1921, 92, I., 200—202.

THE determination of the degree of purity of inactive gases such as nitrogen and the rare gases presents great difficulties. An experimental method for the determination of minute quantities of impurities, e.g., a few thousandths per cent., is based upon the work of Franck and Hertz (Phys. Zeits., 1916, 17, 409) on the ionisation arising from the collisions of molecules and electrons. The current strength of the discharge in a gas is considerably increased when very small quantities of impuri-



ties of an electro-negative character, such as oxygen, nitric oxide, or water, are present. The changes with time of the character of the discharge in nitrogen between a molybdenum point and an aluminium plate, when oxygen and water vapour, respectively, were present as impurities, are described. The pressure of the gas in the discharge vessel was 700 mm. The method enables contamination with oxygen to the extent of  $5 \times 10^{-5}\%$  to be determined. The procedure is complicated in the case of contamination with water vapour, owing to dissociation occurring. With slight modification, the method is applicable to the rare gases. Contamination of nitrogen with from 0.1% of hydrogen and upwards can be accurately determined.

—J. S. G. T.

See also pages (A) 178, *Fusion of carbon* (Ryschkewitsch). 181, *Electrolytic copper* (Grube and Reusz, also Grube). 182, *Electrolytic zinc plant* (Laist and others). 194, *Electrolytic oxidation of alcohols* (Müller and Rius y Miro). 196, *Property of feeble electrical conductors* (Reboul).

#### PATENTS.

*Carbon for electrodes; Apparatus for and method of calcining* — W. Hoopes, Assr. to Aluminium Co. of America. U.S.P. 1,366,457 and 1,366,458, 25.1.21. Appl., 20.5.19.

COKE is calcined in a closed chamber, the raw coke being supplied at the top and calcined coke withdrawn at the bottom. An electric current is passed through an intermediate section of the column of coke to heat it, and a stream of inert gas is forced upwards through the whole column, whereby the calcined coke in the lower section is cooled, the heat uniformly distributed in the intermediate section, and coke in the upper section uniformly preheated. The gas leaving the upper end of the chamber is passed through cleaning and cooling apparatus before being introduced again at the bottom of the chamber. —J. S. G. T.

*Cadmium [terminal] leads for zinc electrodes of galvanic cells.* Deutsche Gasglühlicht A.-G. (Auerger). G.P. 328,639, 15.7.19.

By employing cadmium terminal leads in conjunction with zinc electrodes in galvanic cells, practically no solution of zinc occurs on open circuit. —J. S. G. T.

*Electric accumulators; Process of making negative electrodes for* — A. Pouchain. U.S.P. 1,366,489, 25.1.21. Appl., 13.2.20.

SEE E.P. 150,811 of 1919; J., 1920, 726 A.

See also pages (A) 171, *Electrical purification of gases* (G.P. 325,956). 178, *Copper sulphate* (E.P. 147,689). 179, *Ferrous oxide* (U.S.P. 1,367,409); *Magnesium* (G.P. 328,413). 183, *Welding-electrodes* (E.P. 157,534). 196, *Alcohol* (G.P. 328,342).

## XII.—FATS; OILS; WAXES.

*Lubricating oils.* Lichthardt. See IIa.

*Detergents.* Heermann. See VI.

*Viscosimeter.* Mallison. See XXIII.

#### PATENTS.

*Oil; Extraction of — from oats.* P. Winde. G.P. 327,895, 10.8.19.

AFTER removing 12–30% of husk, the oats are ground to give a yield of 50 to 90% of flour or meal; the residue from the grinding, comprising germ, husk, fibre, and membrane, after being pressed between smooth rolls, is in a convenient form for the extraction of its oil. —D. F. T.

*Detergent.* Chem. Fabr. vorm. Weiler-ter Meer.

G.P. (A) 327,683, 11.6.16, and (s) 327,684, 22.6.18.

(A) FINELY divided, highly absorbent material, such as kieselguhr or clay, is saturated with a hydrocarbon solvent or a chlorine-substitution product, capable of dissolving fats; sodium carbonate and bleaching agents may also be added. The resulting loose powder when mixed with water gives a turbid liquid of great cleansing power, the finely dispersed solid matter preventing the separation of the organic solvents from the water and collecting the dirt. (s) Chlorinated derivatives of aromatic hydrocarbons with from eight to ten carbon atoms, e.g., chlorinated solvent naphtha, and chloroxylene of b.p. 190°–200° C., are used in conjunction with soap and as constituents of fat-free detergents.

—D. F. T.

*Washing and cleansing agents; Production of fat-free* — Elektro-Osmose A.-G. (Graf Schwerin Ges.). G.P. 327,685, 30.7.18.

CLAY or similar material, preferably refined, is mixed with sulphite-cellulose waste liquor and waste liquor obtained in the alkali treatment of vegetable materials, in such proportion as to ensure a fairly permanent suspension of the silicate during washing. The waste liquors, which are conveniently used in a purified and concentrated form, increase the cleansing power of the clay by their ability to form a lather. —D. F. T.

*Soaps and washing materials; Manufacture of* — C. H. Boehringer Sohn. E.P. 139,776, 1.3.20. Conv., 21.11.18.

SEE G. P. 323,804 of 1918; J., 1920, 756 A.

*Alcohols.* G.P. 327,510. See XX.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Turpentine oil; New methods for the examination of* — H. Salvaterra. Chem.-Zeit., 1921, 45, 133–135, 150–151, 158–159.

THE method of Tausz (J., 1918, 554 A), depending on the determination of the acid liberated by the action of mercuric chloride on turpentine in methyl alcohol solution, yields untrustworthy results in that the acid liberated varies with the time of reaction and the origin of the turpentine. A further method of Tausz (*loc. cit.*) in which 20 c.c. of turpentine is shaken with an aqueous solution of 70 g. of mercuric acetate in the presence of a little methyl alcohol, followed by heating for 3 hrs. on the water-bath under a reflux condenser and subsequent steam-distillation, the steam-distilled adulterant being measured in a burette, gave results on turpentine adulterated with solvent naphtha, petroleum benzene, and tetralin agreeing to 1%. The method failed, however, when wood turpentine was present, this product giving a distillate of 10–15%. The determination is most conveniently carried out by a modification of Henderson and Agnew's method (Chem. Soc. Trans., 1901, 95, 291) employing yellow mercuric oxide in acetic acid in place of mercurous acetate. The method of oxidation with a mercurous salt, and weighing the reduced mercurous salt as mercurous chloride (Tausz, *loc. cit.*), is untrustworthy, the results varying with the origin of the turpentine, as also are the methods of Nicolardot and Clement (J., 1910, 501) and Krieger (J., 1916, 746). The bromination method of Allina and Salvaterra (J., 1920, 697 A) for the estimation of petroleum benzene in turpentine has been modified to serve also for the estimation of aromatic hydrocarbons as follows:—12 g. of sodium bromate in 10 c.c. of water is placed in a 500 c.c. Claisen flask and 20 c.c. of the oil under examination added.



Hydrobromic acid (sp. gr. 1.38—1.49) is then added from a burette, 1 c.c. at a time, cooling after each addition, and preferably in the absence of daylight, until the bromine colour persists for 5 mins. The cooling is then arrested, the contents of the flask brought to 30° C., allowed to stand at 20°—25° C. for 15 mins., neutralised with solid sodium carbonate, and decolorised with finely-powdered arsenious acid. The Claisen flask side-tube filled with glass wool and beads is then attached and the contents of the flask steam-distilled until three consecutive 5 c.c. portions of aqueous distillate do not contain more than 0.1 c.c. of oil. A total distillate of 60 c.c. is usually sufficient. In the presence of wood turpentine it is advisable to add an excess of 2 c.c. of hydrobromic acid 10 mins. after the completion of the bromination. Wood turpentine is most conveniently detected by the preliminary application of the Halphen colour test, in which a wine- to blood-red coloration appears when bromine vapour is allowed to act on a carbon tetrachloride solution of the oil in presence of phenol. The bromination method gives a maximum error of 1.25%, but fails in the presence of tetralin as an adulterant, about 70% only of this solvent being recoverable in the distillate. The conversion of turpentine into a halogenated addition product non-volatile in steam forms the basis of the following method for the estimation of adulterants:—10 c.c. of the oil is placed in a 1 litre Claisen flask and a solution of 40 g. of iodine and 52 g. of mercuric chloride in 100 c.c. of alcohol added from a burette with constant cooling. The flask is left in the dark for 6 hrs., strong potassium iodide solution is then added to dissolve the mercuric iodide, the solution decolorised with sodium thiosulphate and neutralised with soda. 200 c.c. is then steam-distilled into a half-litre flask, the neck of which is graduated in 0.1 c.c. divisions and which contains 300 c.c. of water. The volume of distilled oil is read off 30 mins. after the end of the distillation. An apparatus correction, obtained by running blank tests on turpentine adulterated with known and increasing amounts of adulterant, is advisable. Comprehensive test analyses with the method showed a maximum error of 0.5%. The difference between the yields from the mercuric acetate method and either of the halogen addition methods when multiplied by the factor 10 gives approximately the proportion of wood turpentine present in the sample.—A. de W.

*Resin; New synthetic — from benzylaniline.* W. Herzog. Oesterr. Chem.-Zeit., 1921, 24, 16—17.

A mixture of benzylaniline, 9, formaldehyde (40%), 10, water, 20 g., and concentrated hydrochloric acid, 1 c.c., is heated for 1 hr. under a reflux condenser; the liquid is then decanted from the resinous substance which is formed, the latter washed with hot water and dried at 110° C. About 8.5 g. of transparent, light-coloured resin, resembling colophony, is obtained. The resin is soluble in benzene and in trichloroethylene, the solution being suitable for use as varnish; it is soluble in hot, but not in cold, turpentine.—W. P. S.

*Orthodichlorobenzol solvent. Suggestions for its application in the paint and varnish industry and as a constituent of fumigating paints.* H. A. Gardner. Paint Manuf. Assoc., U.S.A., Circ. 116, Feb. 21. 7 pp.

The crude liquid mixture of *p*- and *o*-dichlorobenzene, obtained in the purification of *p*-dichlorobenzene which forms its chief constituent, is miscible with almost all the liquids used in paint and varnish manufacture. In connexion with its use in this direction and independently, it possesses marked insecticidal properties and is of value for combating warehouse insects, clothes moths, cockroaches, and house insects generally.—D. F. T.

*Phenolic hexamethylenetetramine compounds.* M. Harvey and L. H. Baekeland. J. Ind. Eng. Chem., 1921, 13, 125—131.

The hexamethylenetetramine addition products of di-*m*-cresol, di-*p*-cresol, mono-*o*-cresol, triphenol, quinol, resorcinol, and carvacrol have been prepared; they are crystalline substances having no definite m.pt. When heated to near their point of liquefaction they are converted into resinous substances of the bakelite type.—W. P. S.

*Furfural.* Monroe. See XX.

#### PATENTS.

*Sulphide composition [pigment] for painting which resists the action of heat and light.* Y. Nishizawa. E.P. 156,971, 25.11.19.

NEUTRAL or almost neutral organic compounds containing at least two hydroxyl groups, e.g., esters or salts of tartaric or polyhydroxystearic acid, glycol, saccharides, etc. when mixed with zinc sulphide in the proportion of 1 mol. to 20—40 mols. of the sulphide, render it stable to light and heat. Admixture may be effected by kneading the dry pigment with the hydroxy compound or an aqueous solution of the same, by precipitating the zinc sulphide in presence of the hydroxy compound, or by co-precipitation of the sulphide with an insoluble derivative of the hydroxy compound, e.g., zinc tartrate.—A. de W.

*Coke; Drying — for use as pigment.* A. Metzner. G.P. 328,529, 15.8.19.

COKE is quenched with water as it is withdrawn from the retorts, and is charged while still hot into rotating vessels in which it is reduced to a fine dry powder.—L. A. C.

*Zinc white; Preventing the harmful effect of — in mixtures containing colours sensitive to light.* A. Eihner. G.P. 328,648, 14.4.15.

Zinc white is boiled with zinc sulphate, zinc chloride, sodium bicarbonate, or phosphoric acid, and the basic compounds formed, which contain at least 12—14% of insoluble sulphate, chloride, carbonate, or phosphate, are dried and may be heated to low incandescence to increase the covering power.—L. A. C.

*Phenol-aldehyde condensation products; Solutions of —.* G. H. Howse. E.P. 156,896, 13.10.19.

BENZYL alcohol with or without nitrobenzene, and certain aliphatic and aromatic substances, oils, resins, rubber, waxes, cellulose, pigments, etc., is claimed as a solvent for phenol-aldehyde resins for producing a varnish, e.g., benzyl alcohol 3 pts. and nitrobenzene 25 pts., are used with synthetic resin 80 pts., methylated spirit 32 pts., and methyl ethyl ketone 10 pts., all by weight.—A. de W.

*Siccatives; Production of —.* G. Ruth and E. Asser. G.P. 327,374, 18.2.19.

NAPHTHENATES soluble in solvent naphtha and applicable as siccatives are obtained by heating naphthenic acids with oxides or carbonates of chromium or aluminium at 240°—260° C. and expelling any unsaponifiable matter at 350° C., or by precipitation of an alkali naphthenate with an aluminium or chromium salt, washing and drying the precipitate and finally fusing it at 240°—350° C.—D. F. T.

*Siccatives and substitutes for linseed oil varnish or oxidised linseed oil; Production of —.* G. Ruth and E. Asser. G.P. 327,375, 21.6.19. Addn. to 327,374 (cf. supra).

By heating aluminium or chromium naphthenate with magnesia, calcium hydroxide, or zinc oxide at 200° C. or at 160° C. under a pressure of 10 atm.,

products are obtained which alone or mixed with other naphthenates, resins, drying oils, or marine animal oils can be used as siccatives, or as a substitute for linseed oil varnish or oxidised linseed oil.—D. F. T.

*Cellulose- and spirit-varnishes; Method for softening* — G. Ruth and E. Asser. G.P. 327,376, 13.6.19.

For softening cellulose- or spirit-varnishes a castor oil substitute consisting of an ester of naphthenic acid with glycol or glycerol is added.—D. F. T.

*Chloronaphthalenes; Improvement of the technical mixture of — used as a substitute for resin.* Deutsche Conservierungsges. m.b.H. G.P. 327,704, 6.10.17.

The mixture of chloronaphthalenes is submitted, at 215° C., to the action of a current of air or neutral gas for the removal of the volatile constituents; the residue gives rise to practically no hydrogen chloride, and is not injurious to the health of workers.—D. F. T.

*Lead sulphate.* E.P. 157,554. See VII.

*Anti-fouling paints.* E.P. 156,827. See XIXa.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Hevea latex; Coagulation of* — G. Vernet. Bull. Caoutchoucs, Inst. Colon. de Marseille, 1919, 107—121.

The author reviews the general theory of latex coagulation and the methods of effecting the separation of rubber from latex by evaporation, centrifuging, and spontaneous coagulation. For the ordinary methods of coagulation the only generally applicable hypothesis is that coagulation depends on the conversion of the albuminoid matter of the latex into an insoluble condition. A suggestion is made that any advantages possessed by the Brazilian wild rubber may be due to the effect, on the freshly prepared coagulum, of special types of micro-organisms peculiar to the district.—D. F. T.

*Rubber; Action of certain organic accelerators in the vulcanisation of* — G. D. Kratz, A. H. Flower, and B. J. Shapiro. J. Ind. Eng. Chem., 1921, 13, 67—70.

The activity of synthetic nitrogenous organic substances (aniline, its homologues and closely related bases) as accelerators is not proportional to the dissociation constants of the original substances, and, with the exception of closely related series, no definite relationship exists between the activities and dissociation constants. Substances which decompose or dissociate into other substances of acid character, or react to form acid substances, do not accelerate unless a neutralising substance is present. Vulcanisation may be effected by heating rubber in a closed vessel with a concentrated aqueous solution of ammonium sulphides (hydrosulphide and polysulphide).—W. P. S.

#### PATENTS.

*Vulcanisation of rubber, reclaimed rubber and similar mixtures on metals.* Felten und Gnillemaume, Carlswerk A.-G. G.P. 326,541, 10.10.18.

A LAYER of adhesive, viscous material, which is not absorbed by rubber, is interposed between the rubber and the metal; the intermediate- and by-products obtained in the manufacture of synthetic rubber are suitable for the purpose.—D. F. T.

*Vulcanised products; Production of soft and elastic* — Farbenfabr. vorm. F. Bayer und Co. G.P. 326,819, 20.2.17.

VULCANISED products of great strength and extensibility are obtained by adding antimony pentoxide to the raw mixture; the result is a red vulcanisate of greater softness, extensibility, elasticity, and toughness than those obtained with "golden antimony" or "Japan red."—D. F. T.

*Materials resembling rubber or linocryl; Manufacture of* — G. Ruth and E. Asser. G.P. 327,913, 18.2.19.

A FEEBLY alkaline solution of an alkali naphthenate is treated with an aluminium or chromium solution until a salt of hexanaphthenecarboxylic acid ( $\alpha$ -methylcyclopentanecarboxylic acid) begins to be precipitated; in this way the unpleasant odour of the naphthenates is removed. The naphthenates obtained, when heated to 170° C., yield very viscous solutions resembling solutions of rubber, whilst at 300° C. a horny mass is produced which gives mobile solutions in turpentine, pine oil, and solvent naphtha.—D. F. T.

*Rubber; Means [parchmentised or vulcanised paper coverings] to be employed in connexion with the conservation, or storing, of — in sheet form.* J. Y. Johnson. From Diamond State Fibre Co. E.P. 157,673, 30.4.20.

#### XV.—LEATHER; BONE; HORN; GLUE.

*One bath chrome [tanning] liquors and their analysis.* W. Klaber. J. Amer. Leather Chem. Assoc., 1919, 14, 622—626.

COMMERCIAL chrome tanning liquors may contain aluminium, iron, sodium, chlorides, and organic acids in addition to chromium and SO<sub>3</sub>. A portion of the liquor is oxidised with sodium peroxide, boiled, filtered, the Cr<sub>2</sub>O<sub>3</sub> (a) estimated in the filtrate and Fe<sub>2</sub>O<sub>3</sub> (b) in the precipitate. The total acidity (c) is obtained by titrating the boiling solution with N/10 alkali, using phenolphthalein as indicator. A third portion is treated with ammonium chloride and ammonia, filtered, the precipitate washed, dissolved in hydrochloric acid and re-precipitated as before. The precipitate is ignited and weighed as Cr<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, and Al<sub>2</sub>O<sub>3</sub> (d), and the SO<sub>3</sub> is estimated in the combined filtrates. These operations are repeated with a fourth portion substituting ammonium sulphate for the chloride and sulphuric for hydrochloric acid. The chlorides are estimated in the combined filtrates. The total soda (Na<sub>2</sub>O) is obtained by repeating the operations as for the third portion; the combined filtrates are evaporated with excess of nitric acid, ammonium salts decomposed, the residue moistened with sulphuric acid, ignited to constant weight and weighed as sodium sulphate. The "total relative acidity" or ratio of grm.-molecular weights of monobasic acid to grm.-molecular weights of chromic oxide is given by  $8c/a$ . The amount of SO<sub>3</sub> combined with Na<sub>2</sub>O as Na<sub>2</sub>SO<sub>3</sub> is deducted from the total SO<sub>3</sub>, and the difference is "acid SO<sub>3</sub>." The "organic acidity" is the difference between the "total relative acidity" and  $152.2(\text{acid SO}_3)/40.03a$ ;  $\text{Al}_2\text{O}_3 = d - (a+b)$ . Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> are considered to be present as the normal sulphates and SO<sub>3</sub> calculated to be so combined is subtracted from the "acid SO<sub>3</sub>." The difference is SO<sub>3</sub> combined with Cr<sub>2</sub>O<sub>3</sub>. "Basicity" is replaced by "acidity of Cr<sub>2</sub>O<sub>3</sub>," given by the formula:  $-(\text{SO}_3 \text{ combined with Cr}_2\text{O}_3/40.03)/(\text{total Cr}_2\text{O}_3/152.2)$ , which ratio gives the number of (OH) groups of Cr<sub>2</sub>(OH)<sub>3</sub> replaced by acid radicals.—D. W.

*Chrome [tanning] liquor; Effect of concentration of — on the absorption of its constituents by hide substance.* A. W. Thomas and M. W. Kelly. *J. Ind. Eng. Chem.*, 1921, 13, 65–67.

THE curve showing the results of a 2-day test on the absorption of chromium by hide powder sloped steeply downwards after the concentration of the liquor exceeded approximately 18 g. of chromic oxide per litre in a solution of the composition  $\text{Cr}(\text{OH})\text{SO}_4$ , and reached a minimum when the concentration of chromic oxide was 147.5 g. of chromic oxide per litre, this minimum being maintained up to a concentration of 202 g. per litre.

—W. P. S.

*Guyacan.* G. L. Terrasse and J. F. Anthes. *J. Amer. Leather Chem. Assoc.*, 1919, 14, 700–701.

GUYACAN (*Caesalpinia melanocarpa*, Griseb.), of the family *Leguminosae*, is an undersized tree growing in subtropical Argentina, yielding seed pods locally utilised as tanning agents. Analysis of a ground sample by A.L.C.A. methods showed tans 22.53%, non-tans 37.21%, insolubles 28.86%, water 11.4%, sugars (as glucose) 21.1%. Guyacan resembles divi-divi. Its tannin gives a blue-black colour with iron alum, and no precipitate with bromine water. It gives a pale yellow leather which is distinctly plump, soft and pliable.—D. W.

*Tanning extracts; Preparation of — for analysis.* R. H. Forbes. *J. Amer. Leather Chem. Assoc.*, 1920, 15, 21–25.

EXPERIMENTS on the extraction of barks, woods, leaves, etc., show that a percolation method is essential for complete extraction owing to the reabsorption of tanning materials by cellulose, starch, and albuminoids. The percolation method must be modified to suit the material under examination. Continuous extraction at steam heat is suitable for most woody materials, but for canaigre percolation at 50°–55° C. is best. The use of an asbestos filter is necessary to avoid loss of tanning materials. The extractor consists of a Soxhlet fitted with a rubber stopper carrying a 250–500 c.c. funnel filled with water and the stopcock so turned that 500 c.c. of liquid will pass through in 4 hrs. A tank 8"–10" deep has a bottom perforated with holes to receive the rubber stoppers through which the lower ends of the Soxhlet apparatus project. The tank is filled with water at the desired temperature.—D. W.

*Tanning materials; Extraction of — [for analysis] with different extractors.* F. P. Veitch. *Bulletin* 90, U.S. Dept. Agric. *J. Amer. Leather Chem. Assoc.*, 1920, 15, 119–122.

A COMPARISON of results obtained by extracting different tanning materials with three kinds of extractors, viz., Soxhlet, Koch, and continuous (Zulowsky). The continuous form yielded the highest amount of soluble solids. Tannin was highest with the continuous extractor except in the case of sumach, where the Koch apparatus gave the best result.—D. W.

*Tannin; Discussion of the methods for the determination of —.* F. P. Veitch. *Bull.* 90, U.S. Dept. Agric. *J. Amer. Leather Chem. Assoc.*, 1920, 15, 122–127.

ATTEMPTS to estimate tannin by precipitation with ferric salts, aluminium hydroxide, caustic soda, and gelatin, or by neutralisation with calcium hydroxide gave results inferior to those obtained by the hide powder method. This method yields inaccurate results due to a continuous absorption of solids from the solution by the filter paper during the determination of soluble solids and the absorption of colouring matter and other non-tannin matter by the hide powder.—D. W.

*Strap, harness and side leather; Analyses of different tannages of —.* L. M. Whitmore. *J. Amer. Leather Chem. Assoc.*, 1919, 14, 567–576.

THE results of analyses conducted by the U.S. Bureau of Standards on leathers supplied for Government use during the war are given. The petroleum spirit extract and the mineral acidity most frequently failed to come within the specifications. The Government specifications for harness leathers required 15–20% of grease, but the analyses show that it is impossible to comply with specifications which require a definite amount of grease. The grease content in the samples examined varied from 8.1% to 45.7%. A maximum should be stated for leathers sold by weight, and a minimum for those sold by area. Ash, water-soluble matter, glucose, salts, and acidity maxima can be complied with by tanneries run with chemical control. The degree of tannage varied considerably, and 15° should be allowed between the maximum and minimum.—D. W.

#### PATENTS.

*Phenolsulphonic acids; Preparation of condensation products [tanning agents] from —.* F. Hassler. *G.P.* 328,340, 9.12.13.

CONDENSATION products with tanning properties are obtained when phenolsulphonic acid or its homologues are heated with the sulphonic acids of aromatic hydrocarbons, particularly the coal tar hydrocarbons of carbolic, cresotic, and anthracene oils. The sulphonation product, obtained by the action of concentrated sulphuric acid at 100° C. on Russian petroleum, may also be utilised. The sulphonic acids are heated together for some hours at about 100° C. until no further increase is observable in the precipitate obtained from a small test portion on the addition of gelatin. The reaction may be facilitated by the addition of a condensing agent, such as phosphorus trichloride, thionyl chloride, anhydrous aluminium sulphate, or boric acid. The condensation products are soluble in water, give heavy precipitates with gelatin, and violet colorations with ferric chloride. The condensation products of phenolsulphonic acid with benzenesulphonic acid and with naphthalenesulphonic acid both form viscid or almost solid masses, soluble in water to a clear, almost colourless solution.—G. F. M.

*Glue; Manufacture of bone or hide — which remains liquid in the cold.* *Luftfahrzeugbau* Schütte-Lanz. *G.P.* 328,692, 9.12.17. Addn. to 325,246 (*J.*, 1920, 827 A).

FORMALDEHYDE and alum are added to the glue solution in addition to formic acid or a formate, as described in the chief patent. The product does not coagulate for several days, and after coagulation is insoluble in water.—L. A. C.

## XVI.—SOILS; FERTILISERS.

*Soil studies. I. Influence of fertilisers upon the productiveness of several types of soil. II. Influence of fertilisers and plant growth upon soil solubles.* W. H. Jordan. *New York Agric. Exp. Stat. Bull.* 473. Mar., 1920. 27 pages.

THE growth of barley under hot-house conditions on the eleven types of soil examined was influenced very little by the application of either muck or leaf mould. In the first year farmyard manure failed to produce additional growth, but in the second year satisfactory results were obtained. The production with commercial fertilisers was much larger than with stable manure containing the same quantities of nitrogen, phosphorus, and potassium. The increase in yield was not proportional to the increased application of fertilisers.

Slaked lime applied along with stable manure gave very irregular results according to the type of soil. Nitrogen was the only ingredient of the artificial fertilisers which had any marked influence upon the growth of the barley. Under hot-house conditions the soils (one highly productive and one far less productive) appeared to supply sufficient quantities of phosphorus and potassium for luxuriant growth. The application to the soils of soluble compounds of nitrogen, phosphorus, and potassium materially increased the amount of water-soluble substances in the soil. These were utilised by the plants and the reduction in the amount of soluble matter was rapid even before the plants had attained considerable growth. In the later stages the water-soluble material remained practically constant and about equal to that in the soil carrying no crop.—W. G.

*Hydrogen-ion concentration of certain three-salt nutrient solutions for plants.* A. G. McCall and J. R. Haag. *Soil Sci.*, 1920, 10, 481—485.

THE hydrogen-ion concentration of the six types of three-salt nutrient solutions described by Livingston and Tottingham (*Amer. J. Bot.*, 1918, 5, 337—346) have been measured. In general, with any one type, the hydrogen-ion concentration of the solution is a function of the volume-molecular proportion of the dihydrogen phosphate salt present, the sulphates and nitrates apparently playing only a minor part. The types containing  $\text{KH}_2\text{PO}_4$  have a lower hydrogen-ion concentration than those containing either  $\text{Mg}(\text{H}_2\text{PO}_4)_2$  or  $\text{Ca}(\text{H}_2\text{PO}_4)_2$ . The variations in plant growth, with any one type of solution, could not be correlated with differences in the hydrogen-ion concentrations of the solutions. —W. G.

*Carbon bisulphide; Behaviour of — when injected into the soil and its value as a control for the root-form of the woolly apple aphid.* B. R. Leach. *Soil Sci.*, 1920, 10, 421—451.

THE diffusion of carbon bisulphide in the soil is influenced by the soil type, the soil moisture, and the depth and arrangement of the holes where it is introduced. The soil moisture is the limiting factor. Variations in temperature throughout the growing season apparently do not affect the attainment of maximum diffusion. It was not found possible at any time during the year when soil conditions were favourable to apply carbon bisulphide in an amount sufficient to kill the aphid without causing direct injury to the roots and indirect injury to the branches and foliage of the apple trees.—W. G.

*Java indigo plant.* Davis. See IV.

#### PATENTS.

*Fertiliser dryers.* J. P. Hamler, Assee. of P. J. Hamler. E.P. 143,846, 6.5.20. Conv., 28.5.19.

THE apparatus consists essentially of two concentric drums united at their ends only, with a space between them through which steam circulates under pressure. The outer drum has a plain surface; the inner one is of light material and has a corrugated surface; this feature gives increased heating area, allows for contraction and expansion and offers greater resistance to steam pressure, and enables stay-bolts and rivets to be dispensed with. A central shaft passes through the inner drum, and carries arms terminating in blades shaped to conform to the corrugations of the drum; the absence of rivets permits of a very small clearance between the blades and the drum and thereby prevents the formation of a cake, with consequent loss of heat at the surface. The apparatus includes devices for loading and discharging the material; it is also provided with a chamber in communication with

the inner drum for drawing off vapours, a diaphragm across the chamber serving to prevent the escape of small particles of dried material. —W. J. W.

*Lime for use as fertiliser; Manufacture of —.* R. Mandelbaum. G.P. 328,220, 22.12.18.

LIME or lime marl is either heated in a closed furnace, or is heated in an open furnace and then immediately treated with just sufficient water to convert it into calcium hydroxide, and with carbon dioxide.—L. A. C.

*Potassium nitrate etc.* G.P. 306,334. See VII.

#### XVII.—SUGARS; STARCHES; GUMS.

*Sugar; Balance of chlorine during the manufacture of — and the chlorine content of beetroot.* E. Saillard. *Comptes rend.*, 1921, 172, 283—284.

BETROOTS contain on the average 0.016% of chlorine, of which 80% passes into the molasses and 19% into the diffusion residues during the manufacture of sugar. In the plant the petioles contain the highest percentage and total amount of chlorine.—W. G.

*Carbonatation; Influence of the amount of lime used in — on the purity and the calcium content of the clarified juice.* V. Stanek. Z. Zuckerind. Czechoslov., 1920, 45, 53—55.

WHEN, owing to the use of frozen or altered roots, the raw juice contains much more than 0.1% of invert sugar, the organic acids that are formed as the result of the action of the lime on the dextrose and levulose increase to a marked degree the calcium content of the filtered and clarified juice. When such inferior roots are being sliced, the best results in respect of a high quotient of purity and a low calcium content are obtained by using a triple saturation process, the procedure generally being to add 2.3—3.6% of lime to the raw juice, carbonate to an alkalinity corresponding to about 0.10%  $\text{CaO}$ , filter, add 0.55% more lime, carbonate to 0.05—0.06%, filter, carbonate to 0.001—0.003%, and finally boil.—J. P. O.

*Clarification of sugar juice; Increase in the quotient of purity effected by the defecation, sulphitation, and carbonatation processes for the —.* A. Schweizer. Arch. Suikerind. Nederl.-Indië, 1919, 27, 2013—2016. *Int. Sugar J.*, 1921, 23, 106—107.

GENERALLY in Java in determining the increase in the quotient due to clarification, the sample of mill juice is allowed to stand for  $\frac{1}{2}$  hr. in order to allow the suspended matter to subside, and the entangled air to escape, before observing the Brix reading. J. S. de Haan (Arch. Suikerind. Nederl.-Indië, 1919, 27, 1897—1906) suggested that more correct results would be obtained if the juice were filtered through paper or also centrifuged in a Sharples machine, but it is pointed out that the clear liquid thus obtained would not correspond to that entering the factory, since the filter paper would adsorb certain of the impurities, and others would be separated by the high-speed centrifuging. A centrifugal machine may, however, be employed for the removal of the suspended matter, provided its speed is not more than 2000 revs. per min.—J. P. O.

*Decolorising carbon ("Carboraffin"); Stanek's method of using — for the purification of liquors in the refinery.* V. Skola. Z. Zuckerind. Czechoslov., 1920, 45, 89—95.

STANEK'S method of passing the liquor to be treated through a layer of decolorising carbon, in the form of a cake in a filter-press (G.P., 317,449; J., 1920, 380 A), is stated to have the following advantages

compared with the usual treatment with animal charcoal:—The amount of water necessary for sweetening off is at least 10 times less, and the loss of sugar 6 times less; the quotient of purity of the wash-waters falls more slowly; and the duration of filtration is much shorter.—J. P. O.

*Filtering materials consisting of carbon together with other substances [for use in sugar refining].* Daude. Z. Ver. Deuts. Zuckerind., 1921, 13—32.

THE processes of more than 30 German Patents relating to this subject (from G.P. 307 to G.P. 290,656) are described in order.—J. H. L.

*[Sugar and sugar products etc.] Electric oven for the rapid determination of moisture [in —].* G. L. Spencer. J. Ind. Eng. Chem., 1921, 13, 70—72.

THE drying capsule is of metal and has its bottom closed with Monel metal filter cloth; the capsule fits tightly into an opening in an annular channel in the oven, this channel being connected with a vacuum pump. A regulated supply of air is admitted to the oven and is heated by means of an electrical resistance in the oven. By the use of this oven, raw sugar may be dried in 10 mins. and cane bagasse in 30 mins.—W. P. S.

*Maize cobs.* La Forge. See XIXA.

#### PATENT.

*Adhesive; Production of an —.* H. Werner. G.P. 327,377, 29.11.17.

SAWDUST is mixed with water and bacteria cultures, and after a pasty consistency is attained, a part of the mixture is subdivided and placed in vessels containing shavings, casein, and a decoction of seaweed. The subdivision is repeated once or twice more, the various fractions being then re-united with the remainder of the original mixture. After thoroughly mixing, the supernatant liquor is separated from the sediment and concentrated or dried.—D. F. T.

### XVIII.—FERMENTATION INDUSTRIES.

*Saccharase [invertase] preparations; Preparation of highly active —.* O. Svanberg. Z. physiol. Chem., 1921, 112, 104—110. (Cf. J., 1920, 636 A, 795 A.)

It was not found possible to separate invertase from yeast gum by means of filtration through a membrane. This observation coincides with Euler and Fodor's assumption that there is a relationship between the two substances. No improvement in the purity of invertase preparations was observed when they were filtered through a membrane instead of being dialysed.—S. S. Z.

*Yeast manufacture by the aeration method; Replacement of malt combs by ammonium salts in —.* A. Wohl and S. Scherdel. Z. angew. Chem., 1921, 34, 41—45.

In experiments with a 25% sugar solution, containing the necessary inorganic salts, and also organic nitrogen in the form of an extract of malt combs, the authors studied the effect of replacing this organic nitrogen, partially or completely, by equivalent amounts of ammonium phosphate. It is concluded that ammonia cannot replace the whole of the organic nitrogenous nutriment without seriously affecting the reproductive and other activities of the yeast; but it may replace a certain proportion (about 50% in the authors' experiments) without impairing either the quality or quantity

of the yeast crop, and within such limits ammoniacal nitrogen is a complete substitute for an equal weight of organic nitrogen. Even when the seed-yeast is poor in nitrogen and therefore contains no reserve which can be drawn upon for reproduction, the yeast will develop well at the expense of mixtures of organic and ammoniacal nitrogen in suitable proportions.—J. H. L.

*Alcoholic fermentation; Application of the precipitometer, and of an apparatus for the determination of catalase, to the study of the course of —.* K. Schweizer. Bull. Assoc. Chim. Sucr., 1920, 38, 163—171.

THE simplest and most rapid means of measuring yeast growth in the course of fermentation experiments is to centrifuge a few c.c. of the mixed culture, after addition of sodium carbonate solution, in a tube with a narrow graduated end (precipitometer), for 5 mins. at 2000 revs. per min., and to measure the volume of the deposit. An apparatus devised by Koestler (Jahresb. Molkerei Ruetli, 1909) for the determination of catalase, is recommended by the author for the measurement of the gas evolved during fermentation.—J. H. L.

#### PATENT.

*Glycerin fermentation residues.* G.P. 326,728. See I.

### XIXA.—FOODS.

*Milk preservative; Mustard oil as —.* P. Post. Pharm. Wochblad, 1921, 58, 131—138.

ADDITION of mustard oil to milk, in the proportion of 20 drops per litre, retards development of micro-organisms, and samples so treated may be kept unaltered for about four days at normal temperature. The treatment does not affect determinations of specific gravity, fat, acidity, total solids, alcohol test, ash, nitrate, chloride, preservative agents, specific gravity, and refraction of serum, or the polarisation. Mustard oil depresses the freezing-point of milk to an extent dependent on the fat content; the error may be almost entirely eliminated by shaking the milk with 10% of sesame oil. For lactose estimations the filtrate from the asapol treatment (cf. Salomon and Diehl, J., 1919, 301 A) is freed from mustard oil by shaking it three times with petroleum ether; and for albumin estimations the milk must first be mixed with alcohol and the mixture evaporated to half its volume. Catalase cannot be estimated in milk samples which have been treated with mustard oil.—W. J. W.

*Milk; Apparatus for determining the catalase content of —.* A. Machens and F. Cordes. Milchw. Zentr., 1921, 50, 25—27.

TWO bottles are connected at their shoulders by a short length of glass tube fitted with a three-way tap; one bottle is provided with a tubulure at the bottom which is connected with a vertical graduated tube. Water is placed in this bottle so that the level rises to the zero mark of the graduated tube and 15 c.c. of milk is placed in the other bottle; both are closed with stoppers, the three-way tap is turned to put them in communication with the atmosphere, and the whole apparatus is placed for 15 mins. in an incubator at 37° C. Five c.c. of 1% hydrogen peroxide is then added to the milk, the tap is turned to connect the bottles with each other, and the apparatus kept at 37° C. The increase of pressure due to the liberation of gas from the mixture of milk and hydrogen peroxide is measured by the rise of the water level in the graduated tube.—W. P. S.

*Creatine; Simple method of preparation of — from meat extract.* H. Steudel. Z. physiol. Chem., 1921, 112, 53—54.

CREATINE is extracted from meat extract with absolute alcohol on a water-bath and is separated by crystallisation after removing the alcohol by distillation; 1 kg. of extract yields 25—30 g. of air-dry creatine.—S. S. Z.

*Proteins; Demonstration of pyrrole substances in —.* N. Troensegaard. Z. physiol. Chem., 1921, 112, 86—103.

THE author advances a theory that the protein molecule is mainly composed of heterocyclic compounds, and in substantiation of this theory experiments are described in which the presence of pyrrole substances in gliadin and gelatin is demonstrated. (Cf. J.C.S., Mar.)—S. S. Z.

*Proteins; Free amino groups of the —.* S. Edlbacher. Z. physiol. Chem., 1921, 112, 80—85. (Cf. J., 1920, 796 A; 1921, 126 A.)

CASEIN was methylated with dimethyl sulphate and hydrolysed. The N-methyl value was determined in the various fractions. The results obtained were as follows:—N-methyl value of the monoamino acid fraction 0, of the histidine-arginine fraction 7.5, of the lysine fraction 73.4.—S. S. Z.

*Cocoa and its products; Determination of theobromine in —.* R. V. Wadsworth. Analyst, 1921, 46, 32—37.

TEN g. of the sample (previous removal of fat is unnecessary except in the case of cacao beans) is mixed with 3 g. of magnesia and 14 c.c. of water, and the paste is dried partially, but not completely, on a water-bath; after 30 mins. the paste is boiled under a reflux condenser for 30 mins. with 150 c.c. of tetrachloroethane, the solvent, while still hot, decanted through a filter, and the insoluble portion again extracted with tetrachloroethane (120 c.c.). The united extracts are distilled until the volume is reduced to about 5 c.c.; this residual liquid is cooled and 70 c.c. of ether is added. After 18 hrs. the precipitated theobromine is collected, washed with ether, dried at 100° C., and weighed. To the weight is added 0.004 g. to correct for the solubility of the theobromine in the 70 c.c. of ether used. The caffeine present remains in solution in the ether.—W. P. S.

*Corn [maize] cobs; Utilisation of —.* F. B. La Forge. Paper, Dec. 22, 1920, 15—18, 32.

By digesting maize cobs under pressure with water soluble gums and other carbohydrates are obtained which can be used as adhesives or as a source of furfural. The latter is obtained by treatment of the gums with mineral acid (cf. Monroe, p. 194 A). The gums can be used as a substitute for dextrin. Another adhesive is obtained from the residues before the extraction of the cellulose, by treating the cellulosic press-cake with a 1% solution of caustic soda. After the elimination of the adhesives the pulp can be handled in the same manner as wood pulp, but the fibre of the maize cob cellulose is very short, and therefore the use of this product is suggested as a substitute for clay as a filler.—J. C. K.

*Cattle food consisting of hydrolysed sawdust; Preparation and analysis of —.* E. C. Sherrard and G. W. Blanco. J. Ind. Eng. Chem., 1921, 13, 61—65.

WHITE pine sawdust was digested with 1.8% sulphuric acid for 20 mins. under a steam pressure of about 120 lb. per sq. in., sufficient water being also added to the sawdust to raise the ratio of water to

dry wood to about 1.251. The acid liquor was removed as far as possible by centrifuging the mass, the latter extracted five times with hot water, the aqueous extracts and the acid liquor mixed, neutralised with calcium carbonate, filtered, and evaporated under reduced pressure to a syrup. This syrup was added to the extracted material which had been dried in an oven, and the whole again dried until the water-content was less than 15%. The product, ground to pass a 100-mesh sieve, yielded the following results on analysis, the figures in brackets being the corresponding values for the untreated sawdust:—Moisture, 3.88 (6.39); water-soluble, 31.11 (9.21); ether-soluble, 3.80 (4.88); soluble in 1% sodium hydroxide solution, 40.23 (25.39); acetic acid, 0.65 (0.91); pentosan, 5.66 (9.86); methylpentosan, 2.50 (2.83); cellulose, 37.90 (54.45); crude fibre, 49.31 (60.81); reducing sugars, 16.20 (none); ash, 3.35 (1.52)%. The cellulose obtained from the digested wood was converted into a viscous semi-transparent mass when treated with 17.5% sodium hydroxide solution, and thus differed from the cellulose from the untreated sawdust, over 50% of this remaining unaffected by the alkali.—W. P. S.

*Disinfecting cereals.* Piutti and Mango. See XIXB.

*Inositol phosphoric acids.* (1) Posternak. (2) Anderson. See XX.

#### PATENTS.

*Lupins; Removal of the bitterness of — by means of a saline acid solution.* C. F. Hildebrandt and B. Rewald. G.P. 327,368, 6.7.18.

THE lupins are steeped in water for several hours; a small quantity of a saline acid solution is then introduced and the soaking continued for several hours longer. The treated material is washed with water and finally extracted with pure water.—D. F. T.

*Evaporation of liquids [milk etc.]; Process of and apparatus for heat treatment or —.* J. F. Ruff. E.P. 157,514, 6.10.19.

SEE U.S.P. 1,354,342 of 1920; J., 1920, 733 A.

*Oil from oats.* G.P. 327,895. See XII.

*Synthetic apple oil.* U.S.P. 1,366,541. See XX.

### XIXB.—WATER PURIFICATION; SANITATION.

*Cereals; Use of chloropicrin in the disinfection of —.* A. Piutti and A. Mango. Giorn. Chim. Ind. Appl., 1920, 2, 677—682.

CHLOROPICRIN, applied in the proportion of 20 c.c. per cub.m. of space, and allowed to act for at least a week at a temperature not lower than 15° C., serves as an efficient parasiticide. The grain store should be sealed hermetically and should be away from inhabited localities, and workmen engaged in handling the chloropicrin, which is sprinkled directly on to the grain, should be furnished with masks impregnated with a solution of 5 pts. of glycerin, 20 pts. of sodium sulphite, and 5 pts. of sodium carbonate per 100 pts. of water. The grain should be turned 24 hrs. after the store has been opened. No injurious effect is produced on the grain or on the flour made therefrom, and the panification of the flour with yeast remains unaffected. The germination is, however, lowered by about one-third, so that treatment with chloropicrin should not be applied with grain to be used as seed. The treatment is cheaper than that with carbon bisulphide, and, furthermore, all danger of fire is avoided.—T. H. P.



**Oligodynamics of silver.** \* R. Doerr. Biochem. Zeits., 1921, 113, 58—69. (Cf. J., 1920, 702 A.)

SILVER which has been inactivated by heating to redness regains its bactericidal activity when exposed to the atmosphere. If it is placed in melted paraffin this regeneration does not take place. The oligodynamic action is due to the formation of soluble silver compounds on the surface of the metal through the oxidising influence of the atmosphere. A certain surface of silver imparts a definite oligodynamic action to a volume of water, and this action diminishes owing to the fact that the soluble substances on the surface diffuse; only by the introduction of a fresh piece of silver with a surface covered with the soluble substances can the oligodynamic action be renewed. The metallic silver does not go into solution. Typhoid, paratyphoid A, and paratyphoid B form colonies on agar plates at a certain distance from the silver and behave under the conditions almost like *B. coli*. On the addition of lactose, on the other hand, the difference in the distance from the silver at which the typhoid groups and *B. coli* will form colonies is more marked and the author utilises this phenomenon in order to isolate *B. coli* from such a mixture of organisms. In some instances the author succeeded in suppressing the growth of *B. coli* in the mixture by means of the oligodynamic action of silver in a fluid medium.—S. S. Z.

**Toxic gases; Use of alkaline polysulphides for neutralising certain** —. Desgrez, Guillemard, and Labat. Comptes rend., 1921, 172, 342—344.

USING a liquid containing 240 g. of sodium polysulphide and 140 c.c. of soap-boilers' lye per litre, diluted just before use by addition of 10 l. of water, the authors have determined the volume of liquid to be sprayed into 20 cub. m. to neutralise given amounts of different toxic gases. The composition of the spray fluid varies with the type of commercial polysulphide used.—W. G.

**Copper; Non-toxicity of** — for mildew. G. and G. Villedieu. Comptes rend., 1921, 172, 335—336.

IT is shown that, in a solution of ammonium carbonate (1 in 10,000 or 1 in 8000), saturated with basic copper carbonate, *Phytophthora infestans* grows and the conidia evolve zoospores, which in their turn continue to evolve and grow.—W. G.

#### PATENTS.

**Anti-fouling paints; Poison compositions for** — and for other purposes. J. A. Lillie. E.P. 156,827, 14.4.19.

THE juice of the West African upa, iza, or ipa shrub (*Alchornea cordifolia*, Muell.-Arg.), especially the juice of the berries or the dried and pounded berries, is used alone or in suitable combination as an anti-fouling paint for ships' bottoms or for other purposes.—A. de W.

**Sheep and cattle dips and washes.** I. McDougall and F. Howles. E.P. 157,527, 11.10.19.

A NON-IRRITANT sheep dip which is unaffected by the hardness of the water used for solution is prepared by heating 35 pts. of arsenious oxide with 65 pts. or more of glycerin, and dissolving 1 lb. of the product, which is a viscous white fluid, in 10 galls. of water.—L. A. C.

**Ultra-violet rays; Apparatus for treating liquids by** —. J. C. Pole, Assr. to The Chemical Foundation, Inc. U.S.P. 1,367,090, 1.2.21. Appl., 28.3.16. Renewed 21.5.20.

A NUMBER of liquid containers are provided with passages of varying cross-section and a narrow channel constructed of a material capable of transmitting ultra-violet rays. An electric lamp adjoining the containers supplies rays both to the containers and to the channel.—L. A. C.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Cocaine; Purification of** —. L. Reutter de Rossmont. Bull. Sci. Pharmacol., 1920, 27, 359—363, Chem. Zentr., 1921, 92, I., 90.

FOUR processes for the purification of crude Brazilian or Argentine cocaine from hygrine, isotropinecocaine, isococaine, and cinnamylcocaine were examined. By dissolving the crude material in hot petroleum ether, pouring off the solution after cooling from the oily resinous residue, precipitating the cocaine with dry hydrogen chloride, dissolving the hydrochloride in water, precipitating isococaine etc. by ammonium carbonate, and then adding ammonia, nearly chemically pure cocaine in 73—75% yield is obtained. By dissolving the crude product in amyl alcohol at 50°—60° C., filtering the cold solution, extracting with dilute hydrochloric acid, and then treating further as in the first method, a yield of 73% is obtained. A 70—73% yield of pure cocaine is obtained by dissolving the crude cocaine in dilute hydrochloric acid, shaking out with amyl alcohol in presence of ammonium carbonate, and treating the aqueous layer as in the first method, the cocaine obtained being recrystallised from a mixture of equal parts of alcohol, ether, and ligroin. By dissolving the crude material in hot naphthalenesulphonic acid, acetic acid, benzoic acid, or oxalic acid, preparatory to treatment by the first method, a yield of 76—79% is obtained. The above yields can be increased by working up the resinous residue to ecgonine and synthetic cocaine.—G. F. M.

**Theobromine and caffeine; Reaction to distinguish** —. M. Malm. J. Pharm. Chim., 1921, 23, 89—91.

THE difference between the behaviour of theobromine bismuthic iodide and the corresponding compound of caffeine towards the reducing action of hydriodic acid may be used to differentiate these two substances. The test is conveniently performed as follows:—0.05 g. of theobromine or caffeine is shaken with 10 c.c. of water and 0.5 c.c. of freshly prepared potassium bismuthic iodide solution. In each case an orange-coloured precipitate is formed, and 5 drops of a 10% tincture of iodine (not freshly prepared, so that it may contain a small proportion of hydriodic acid) is added. The colour of the caffeine bismuthic iodide precipitate changes to a bright red, whilst the theobromine precipitate becomes brown in 15 mins., and dark chocolate brown in less than 30 mins. owing to reduction. A dilute solution of hydriodic acid, containing not more than 1% of hydrogen iodide, may be used instead of the tincture of iodine.—G. F. M.

**Lycorine; Occurrence of** — in the family of the Amaryllidaceae. K. Gorter. Bull. Jard. Bot., 1920, 2, 331—334. Chem. Zentr., 1921, 92, I., 92.

IN addition to the already known sources of lycorine in the Amaryllidaceae, the alkaloid also occurs to the extent of 0.9% in the fresh tubercles of *Amaryllis Belladonna*, 0.3% in the fresh roots of *Clivia miniata* Benth., 0.9% in *Sprekelia formosissima* Herb., in *Cooperia Drummondii* Herb., and in the fresh tubers of *Cyrtanthus pallidus* Sims. The alkaloids of the first and fourth of the above plants designated by Fragner (J., 1891, 847) belamarin and amaryllin, and the alkaloid resembling veratrine isolated by Molle (Jahresber. Ph., 1903, 27) from *C. miniata* are all identical with lycorine. The solubility of lycorine in chloroform and ether observed by Fragner applies only to the amorphous variety, crystalline lycorine being insoluble.—G. F. M.



*Glucosides.* VIII. *Glycyrrhizin.* P. Karrer, W. Karrer, and J. C. Chao. *Helv. Chim. Acta*, 1921, 4, 100—112.

GLYCYRRHIZIC acid, from *Glycyrrhiza glabra*, is free from nitrogen, the supposed presence of the latter being probably due to the formation, during the combustion, of methane. Glycyrrhetic acid, prepared by hydrolysing potassium glycyrrhizate, has the formula  $C_{42}H_{62}O_{16}$  or possibly  $C_{44}H_{64}O_{16}$ . Potassium glycyrrhizate undergoes complicated hydrolysis, the final products being glycyrrhetic acid (1 mol.) and glycuronic acid lactone (2 mols.). (Cf. J.C.S., Mar.)—T. H. P.

*Hiptagin*, a new glucoside from *Hiptage madagascariensis*. K. Gorter. *Bull. Jard. Bot.*, 1920, 2, 187—202. *Chem. Zentr.*, 1921, 92, I, 91—92.

HIPTAGENIN,  $C_{15}H_{21}N_2O_5 \cdot \frac{1}{2}H_2O$ , obtained by extraction of the roots with acetone or ethyl acetate, forms fine silky needles, m.p.  $110^\circ C$ , insoluble in ether, chloroform, hydrocarbons, etc., and not hydrolysed by emulsin. By the action of dilute acids or alkalis dextrose is formed and an intermediate product, hiptagenin, which was not isolated but which gave tartaric acid on further treatment with acids, and ammonia and hydrogen cyanide with alkalis. From these and other considerations it is concluded that hiptagenin is an iso-oxazole derivative, and hiptagin its glucoside ether. —G. F. M.

*Inositol hexaphosphate.* S. Posternak. *Helv. Chim. Acta*, 1921, 4, 150—165.

THE results previously obtained (J., 1919, 695 A) are confirmed, and Anderson's criticisms on the author's work refuted (J., 1920, 673 A). Owing to the energetic retention, by inositol hexaphosphate and most of its salts, of 3 mols. of water which it is impossible to expel, even partially, without decomposing the compounds, the author proposes to express the formula in the form,  $C_6H_{10}O_6 \cdot P_6 \cdot 3H_2O$ . —T. H. P.

*Inositol phosphoric acids.* I. *Synthesis of phytic acid.* II. *Composition of inositol phosphoric acid of plants.* R. J. Anderson. *New York Agric. Exp. Stat. Tech. Bull.* 79, May, 1920. 22 pages. (Cf. J., 1920, 673 A.)

THE author attempted to repeat Posternak's synthesis of phytic acid by heating together inositol, phosphoric acid, and phosphorus pentoxide (J., 1919, 695 A), but the only product which he could isolate corresponded to an inositol ester of pyrophosphoric acid having the formula  $C_6H_{10}O_6 \cdot P_2$ . Its properties and reactions differ in several important particulars from those of phytic acid. It gives a silver salt  $C_6H_8O_6 \cdot P_2 \cdot Ag_2$  and a barium salt  $C_6H_8O_6 \cdot P_2 \cdot Ba_2$ . Further investigations of the inositol phosphoric acid of wheat bran, including analyses of the carefully purified barium salts, lead to the conclusion that the phytic acid of plants is inositol hexaphosphoric acid,  $C_6H_8O_6 [PO(OH)]_6$ . —W. G.

*D. M. (Diphenylamine arsenious chloride).* A. Contardi. *Giorn. Chim. Ind. Appl.*, 1920, 1, 11—26.

WHEN the double compound of aniline hydrochloride and zinc chloride (1 mol.) is heated with aniline (2 mols.), ammonia is liberated and diphenylamine formed:  $ZnCl_2 \cdot 2C_6H_5 \cdot NH_2 \cdot HCl + 4C_6H_5 \cdot NH_2 = 2NH_3 + 2(C_6H_5)_2 \cdot NH + ZnCl_2 \cdot 2C_6H_5 \cdot NH_2 \cdot HCl$  and  $ZnCl_2 \cdot 2C_6H_5 \cdot NH_2 \cdot HCl + 2NH_3 = ZnCl_2 \cdot 2C_6H_5 \cdot NH_2 \cdot HCl + 2NH_3$ . The preliminary preparation of aniline hydrochloride is unnecessary, addition of zinc and ammonium chlorides to heated aniline yielding diphenylamine; after a small quantity of zinc chloride has been added and the reaction started, a mixture of zinc oxide and ammonium chloride may be introduced. Successive additions of aniline

may be made and the residual mass fractionally distilled in a vacuum, a yield of 90% of diphenylamine being obtainable. A plant designed to produce about 500 kg. of diphenylamine per 48 hrs. is described. For the preparation of diphenylamine arsenious chloride, known as D.M. or adamsite, fused diphenylamine (1 mol.) is treated with hydrochloric acid (sp. gr. 1.18) (1.1 mol.), the mass heated and stirred until almost free from water, and the hydrochloride (2 mols.), either moist or after further drying in a desiccator at  $50^\circ$ – $60^\circ C$ , mixed with arsenic trioxide (1 mol.) and fused in a covered vessel provided with a wide efflux tube; when fusion has been maintained for 4 hrs., the temperature rises to  $200^\circ C$  and the reaction is finished, a 95% yield being obtained. A plant for producing 6 tons of D.M. per 24 hrs. is described.—T. H. P.

*Furfural; Preparation and technical uses of —.* K. P. Monroe. *J. Ind. Eng. Chem.*, 1921, 13, 133—135.

FIVE hundred g. of maize cob adhesive (a concentrated aqueous suspension of gums rich in pentosan) is steam distilled with a mixture of 150 c.c. of sulphuric acid (sp. gr. 1.84) and 500 c.c. of water, 4 l. of distillate being collected. The distillate is again distilled through an efficient fractionating column; the furfural distils over in the first portion of the distillate and is separated from the aqueous portion of the distillate and once more distilled. This yield is about 26% of the solid substance contained in the original material. Furfural reacts with alkali sulphides and hydroxides to yield two direct dyes, and gives resinous condensation products with phenols. By the interaction of furfural alone or with acetons in the presence of alkalis soluble resins are obtained which may be useful in the varnish industry. (Cf. La Forge, p. 192 A).—W. P. S.

*Amines; General method of preparation of — from aldehydes or ketones.* G. Mignouac. *Comptes rend.*, 1921, 172, 223—226.

THE aldehyde or ketone is dissolved in the requisite amount of an 8% solution of ammonia in absolute alcohol, finely divided nickel is added as a catalyst, and the mixture is shaken in hydrogen at the ordinary temperature and pressure until the absorption of hydrogen ceases. The amine is then isolated in the usual manner.—W. G.

*Amines; Catalytic preparation of secondary — and attempts at alkylation of these bases.* A. Mailhe. *Comptes rend.*, 1921, 172, 230—233.

SCHIFF's bases of the type  $RCH:NR'$  may readily be converted into the corresponding secondary amines in good yields by suspending in the liquid at  $170^\circ C$  a little finely divided nickel and passing in a current of hydrogen. When the vapour of a secondary amine is mixed with the vapour of methyl or ethyl alcohol and passed over alumina at  $380^\circ$ – $400^\circ C$ , the corresponding tertiary amine is not obtained, but the molecule is decomposed. Thus benzylphenylamine and methyl alcohol give toluene and methyl- and dimethylaniline.—W. G.

*Electrolytic oxidation of methyl and ethyl alcohols in alkaline solution. Electrolytic formation of methane.* E. Müller and A. Rius y Miró. *Z. Elektrochem.*, 1921, 27, 54—57.

By the electrolytic oxidation of methyl alcohol in aqueous caustic soda solution at a platinum anode, small quantities of hydrogen are formed at ordinary temperatures. The most favourable strength of alkali is about 4N, and variations in the current density between 0.006 and 0.018 amp. per sq. cm. have little influence. The best yield of hydrogen obtained was about 11.0% of the theoretical. Ethyl alcohol under similar conditions gives a mixture of hydrogen and methane. Attempts to obtain

hydrogen by the electrolysis of formaldehyde or formic acid at platinum anodes failed, so the formation of these as intermediate sources of hydrogen in the oxidation of methyl alcohol is excluded. The hypothesis is put forward that an oxy-alcohol is formed primarily and that this decomposes into hydrogen and formaldehyde. In the case of ethyl alcohol, the oxy-alcohol formed can break down to give hydrogen and methane, or ethane, but only traces of ethane, at most, are formed. (Cf. J.C.S., April.)—E. H. R.

*Creatine.* Steudel. See XIXA.

*Maize cobs.* La Forge. See XIXA.

#### PATENTS.

*Lobelia inflata*; Process for obtaining the active ingredient of —. C. H. Boehringer Sohn. E.P. 145,621, 29.6.20. Conv., 4.4.14.

THE plant when extracted with alcohol, ether, or benzene, and the extractive matter purified by transference to dilute acid solution and back to ether, furnishes a crude lobeline which actually contains at least three alkaloids— $\alpha$ -lobeline,  $C_{11}H_{17}NO_2$ , colourless crystals, m.p.  $120^\circ C.$ , having the characteristic physiological action of *Lobelia inflata*;  $\beta$ -lobeline, non-crystalline and characterised by a hydrochloride sparingly soluble in water; and  $\gamma$ -lobeline, also non-crystalline, and forming non-crystalline salts. The  $\alpha$ - and  $\beta$ -bases form sparingly soluble mercurous double salts which may serve for the purification of the alkaloids. For the isolation of pure  $\alpha$ -lobeline the crude alkaloidal mixture obtained as above is dissolved in dilute hydrochloric acid; on standing,  $\beta$ -lobeline hydrochloride crystallises out, whilst  $\alpha$ -lobeline hydrochloride is extracted from the solution by repeated agitation with chloroform. After agitating with soda solution, the chloroform solution is evaporated to dryness; the residue crystallises after keeping a short time, and the base may be purified by recrystallising from ether.—G. F. M.

*Butyl p-aminobenzoate*; Manufacture of normal —. Soc. Chim. des Usines du Rhône. E.P. 148,743, 12.5.20. Conv., 30.7.19.

*n*-BUTYL *p*-aminobenzoate, prepared by the esterification of *p*-aminobenzoic acid with *n*-butyl alcohol in presence of gaseous hydrogen chloride, or by esterification of *p*-nitrobenzoyl chloride or *p*-nitrobenzoic acid with *n*-butyl alcohol by the usual methods, and reduction of the resulting *n*-butyl *p*-nitrobenzoate, is a crystalline substance, m.p.  $59^\circ C.$ , b.p.  $173^\circ$ — $174^\circ C.$  at 8 mm. pressure. It is only very slightly soluble in water. *n*-Butyl *p*-nitrobenzoate, a substance not hitherto described, crystallises in lamellæ. It melts at  $35^\circ C.$ , and boils at  $160^\circ C.$  at 8 mm. pressure.—G. F. M.

*Acetic acid*; Manufacture of —. H. Dreyfus. E.P. 156,916, 17.10.19.

IMPROVED results in the oxidation of acetaldehyde to acetic acid are obtained by employing as accelerating catalyst about 0.25% of ferric oxide specially prepared by precipitation from a ferric salt solution and subsequent calcination at a temperature between  $310^\circ$  and  $500^\circ C.$ , the catalytic activity reaching its maximum when the oxide has been heated to above  $400^\circ C.$  This catalyst can also be used advantageously in conjunction with sodium acetate in proportion not greater than double that of the ferric oxide. The operation is conducted with liquid acetaldehyde in a closed vessel provided with a high velocity stirrer, and if pure oxygen is employed the gas is absorbed as fast as it is introduced, and the vessel must be cooled to keep the temperature down to about  $10^\circ C.$  With air the absorption is slower.—G. F. M.

*Diethyl sulphate*; Preparation of —. N. V. Sidgwick, S. G. Preston, and A. Boake, Roberts and Co., Ltd. E.P. 157,578, 30.10.19.

DIETHYL sulphate is obtained by passing ethylene, or ethylene mixed with an inert gas, into sulphuric acid of preferably about 100% strength, or into a solution of ethyl hydrogen sulphate in sulphuric acid, prepared by any other means such as by the action of ethyl alcohol on sulphuric acid. The best results are obtained at a temperature of about  $75^\circ C.$ , using ethylene in large excess. The diethyl sulphate is isolated from the reaction mixture by extraction with an immiscible solvent such as benzene or ligroin, or in any other suitable manner, and is purified by distillation under reduced pressure, the yield being at least 80% by weight of the reaction product. If desired the reaction mixture can be used as an ethylating agent without further treatment.—G. F. M.

*Sacchorin*; Process for producing —. J. Bebie, Assr. to Monsanto Chemical Works. U.S.P. 1,366,349, 25.1.21. Appl., 7.9.20.

*o*-TOLUENESULPHAMIDE is oxidised below  $75^\circ C.$  by means of a mixture containing chromic acid dissolved in sulphuric acid of at least 50% strength by weight.—L. A. C.

*Apple-oil*; Synthetic —. F. B. Power and V. K. Chesnut. U.S.P. 1,366,541, 25.1.21. Appl., 19.7.20.

THE oil consists of the amyl esters of formic, acetic, caproic, and caprylic acids, together with acetaldehyde.—L. A. C.

*Urea*; Evaporating solutions of — [prepared from calcium cyanamide]. A.-G. für Stickstoffdünger. G.P. 299,132, 19.3.16.

A WEAK acid or acid salt, e.g., 1% of phosphoric acid or superphosphate, is added to the solution during evaporation to hinder loss of nitrogen and to prevent reaction between the sulphur compounds present in the solution and metal parts of the apparatus and consequent contamination of the urea with metallic sulphides.—L. A. C.

*Urea*; Manufacture of — [from calcium cyanamide]. A.-G. für Stickstoffdünger. G.P. 301,262, 22.3.16.

A CATALYST, such as manganese dioxide, is added to the paste obtained by treating a suspension of calcium cyanamide in water with carbon dioxide. On heating the mixture at  $85^\circ C.$  for  $\frac{1}{2}$  hr. the whole of the cyanamide is converted into urea. The catalyst is advantageously prepared *in situ* by adding potassium permanganate to the paste and then a reducing agent such as hydrogen sulphide, thiourea, or sulphur dioxide, or by the action of calcium carbonate on ferric chloride or sulphate.—L. A. C.

*Urea*; Manufacture of —. Badische Anilin und Soda Fabr. G.P. 301,751, 30.11.16.

IN heating under pressure compounds of ammonia with carbon dioxide for the manufacture of urea, action on the metal parts of the apparatus is prevented by excluding oxygen and/or using excess of ammonia.—L. A. C.

*Ointment bases*; Manufacture of —. Farbenfabr. vorm. F. Bayer und Co. G.P. 301,665, 27.6.16.

NAPHTHALENE, or its derivatives, e.g., methyl-naphthalene, is condensed with aralkyl halides, e.g., benzyl or xylol chloride, by heating in the presence of iron or iron compounds. The oily hydrocarbons produced if mixed with paraffins, spermaceti, or the like, yield soft and stable ointment bases.—L. A. C.

**Alcohols; Manufacture of high molecular** — G. Schicht A.-G. G.P. 327,510, 16.5.16. Conv., 12.4.15.

ALKYL derivatives of higher oxidised compounds than the required alcohols are heated with aqueous alkali hydroxides and alcohols. Stearic acid ketone (2 mols.), aqueous sodium hydroxide (1 mol.), and ethyl alcohol (2 mols.) when heated together for 3–6 hrs. at 200°–300° C. yield acetic acid and 1.8-pentatriacontanol,  $C_{37}H_{76}OH$ , m.p. 93°–94° C. Alcohols produced from resin acids are viscous liquids. A small part of the product is often hydrolysed to ethers or ethylene compounds. Aldehydes or peroxides produced together with acids and alcohols by the incomplete oxidation with air or oxygen of paraffin hydrocarbons are converted by treatment with aqueous alkalis into the corresponding alcohols; wax alcohols are obtained in this way from pentatriacontane.—L. A. C.

**Berberine, cotarnine, and hydrastinine; Preparation of double salts of** — M. Freund. G.P. 328,101, 14.12.18.

WELL-DEFINED double salts are produced when salts of berberine with polybasic acids are brought into solution with similar salts of cotarnine and/or hydrastinine, and crystallised together, or when the acid is neutralised with a mixture in suitable proportions of berberine with cotarnine and/or hydrastinine. These double salts have a physiological action different from that of the individual constituents. The following salts are described:—Double sulphate containing  $\frac{1}{2}$  equiv. each of berberine and cotarnine, yellow needles, m.p. 190° C.; a similar double salt containing hydrastinine instead of cotarnine, m.p. 172°–173° C.; a complex sulphate containing  $\frac{1}{2}$  equiv. of berberine, and  $\frac{1}{2}$  equiv. each of hydrastinine and cotarnine, yellow needles soluble in water and alcohol, m.p. 160° C.; and a double salt prepared from phosphoric acid (1 mol.) and 1 mol. each of hydrastinine and berberine, which decomposes at 225° C.—G. F. M.

**Hydrocotarnine; Preparation of derivatives of** — M. Freund. G.P. 328,102, 8.4.19.

By the action of a mixture of acetic anhydride and sulphuric acid on hydrocotarnine for 30 mins. at 30° C. a hydrocotarninesulphonic acid,  $C_{17}H_{19}O_6NS$ , is produced. The substance has both acidic and basic properties, is soluble in hot water, insoluble in organic solvents, and melts at 292° C. If the treatment of hydrocotarnine with the acid reagent is carried out at 80°–85° C. a crystalline base,  $C_{17}H_{19}O_6N$ , m.p. 130° C., is produced. It is soluble in benzene, chloroform, and boiling alcohol, and is an acetylhydrocotarnine (6.7-methylenedioxy-8-methoxy-5-acetyl-2-methyltetrahydroisoquinoline). Both the above compounds possess therapeutic value.—G. F. M.

**Ethyl alcohol; [Electrolytic] manufacture of** — from acetaldehyde. Chem. Fabr. Griesheim-Elektron. G.P. 328,342, 28.10.17.

ACETALDEHYDE is passed into an acid solution in the cathode chamber of an electrolytic cell under such conditions that a low concentration, i.e., less than 10%, of acetaldehyde is maintained in the solution.—L. A. C.

**Brometellin; Preparation of** — P. Bergell. G.P. 330,256, 2.8.18.

VITELLIN from egg-yolk is treated with bromine in the presence of an indifferent solvent, such as carbon tetrachloride, benzene, etc., and the resulting product dried at a low temperature. It is a bright yellow substance, insoluble in water, organic solvents, or dilute acids, but soluble in dilute alkalis. It contains 10% Br, which is com-

pletely removed as sodium bromide on prolonged boiling with sodium hydroxide. It serves as a nutrient and sedative, especially in cases of undernourishment where the nerves are also affected.

—B. V. S.

**Metallic hydrosols.** G.P. 326,655. See X.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Electrical conductors; New property of substances which are feeble** — G. Reboul. Comptes rend., 1921, 172, 210–212.

IN explanation of the phenomena previously recorded (J., 1921, 62A), it is suggested that at the portions of the paper or other material where discontinuities occur, the potential differences are sufficient to produce disruptive discharges either in the conductor itself or in the surrounding medium. The discharges thus produced are accompanied by the emission of a radiation which is very readily absorbed and which, like ultra-violet rays and X-rays, though invisible to the eye, is capable of acting on a photographic plate.—W. G.

### PATENTS.

**Dye transfer printing from photographic negatives.** W. F. Donisthorpe, and Dye Impression Photos, Ltd. E.P. 158,021, 30.9.19.

IN the process described in E.P. 13,874 of 1907 (J., 1908, 140), by which a negative is selectively hardened by treatment with a solution of a vanadium salt, dyed, and the dye then transferred, by contact, to a prepared paper, the staining of the hardened negative is made quite regular by the addition of a suitable proportion of acid. The bath recommended is a mixture of equal parts of a 10% solution of vanadium oxalate crystals (vanadium oxalate 6.5%, excess oxalic acid 73.5%, water 20%) and 2% solution of potassium ferricyanide; for dense negatives the solution may be further diluted or the potassium ferricyanide omitted. The impermeability to the dye is so much greater with the film hardened with vanadium salts than with gelatin hardened with alum, formalin, or the like, that the latter may be used for reception of the dye picture. This allows quick drying of the print at high temperatures, thus reducing spreading to a minimum, and also allows the use of cheap paper bases, baryta-coated paper, art printing paper, etc. without any further preparation.—B. V. S.

**Colouring photographs; Process for** — Ges. für angew. Photographie m.b.H. G.P. 323,618, 25.9.18.

THE picture is formed in relief either by the use of a developer which hardens the colloid film in proportion to the amount of image formed or by the use of a normal developer followed by a hardening solution which has the same selective action. The unhardened parts may then be washed away before staining, or the film may be treated with dyes which stain the unhardened but not the hardened portions.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

**Mercury fulminate; Reactions of** — with sodium thiosulphate. F. H. and P. V. Dupré. Analyst, 1921, 46, 42–43.

AMMONIA is one of the final products of the reaction between mercury fulminate and sodium thiosulphate, and the equation  $Hg(CNO)_2 + 2Na_2S_2O_3 + 2H_2O = HgS + 2Na_2SO_4 + NH_4CNS + CO_2$  is probably correct for the ultimate products; the presence of ammonium thiocyanate is, however, difficult to explain if the second phase of the reaction given by

Marshall (*cf.* Heaven, J., 1918, 143 r) is correct. Boric acid may be used in place of potassium iodide in the determination of mercury fulminate by the thiosulphate reaction, since it is equally efficient in preventing loss of alkalinity due to secondary reactions. The precautions to be adopted in the destruction of waste fulminate by means of thiosulphate solution are described.—W. P. S.

#### PATENTS.

*Explosives.* J. H. Babcock, Assr. to Hooker Electro-Chemical Co. U.S.P. 1,366,048, 18.1.21. Appl., 9.5.18.

A CRYSTALLINE explosive (m.p. 65°–85° C.) contains dinitro-para- and ortho-dichlorobenzene, the former compound preponderating.—W. J. W.

*Potassium chlorate; Process of purifying — for explosives manufacture.* K. W. Jurisch and H. von Schleinitz. G.P. 300,714, 11.4.15.

IN order to remove chloride from potassium chlorate crystals, these are first dried, then coarsely ground, and washed.—W. J. W.

### XXIII.—ANALYSIS.

*Optical pyrometry; Comparison of monochromatic screens for —.* W. E. Forsythe. J. Opt. Soc. Amer., 1921, 5, 84–95.

DETAILED results of an investigation are given concerning the suitability of glass screens of different colours in monochromatic optical pyrometry. Two thicknesses each of blue violet glass, Corning green glass, and Jena red glass were employed, the respective total thicknesses being 3.9 mm., 5.2 mm., and 7.8 mm. The transmission curves and the respective effective wave lengths for the various glasses at different temperatures are given. Using the different coloured glasses as screens, determinations were made by different observers of temperatures ranging from 1245° K to 2154° K, employing an optical pyrometer of the disappearing-flame type. The results indicate that throughout the range of temperatures employed, red and green screens are to be preferred to a blue screen. There was very little to choose between the red and green screens. For temperatures below 1200° K, a red screen is the best. For temperatures of about 1400° K, readings can be made with either a red or green screen with accuracy of setting somewhat greater than 0.1%. The error of setting in the case of the blue screen was about 0.3–0.4%. The following reasons are advanced for the choice of a red screen. With a red screen, readings can be made at a lower temperature than with green or blue screens. The rate of colour change with wave length is much less in the red portion of the spectrum than in the green. The spectral transmission of the red screen ends with a steeper slope than does that of the green screen. When employing a rotating sector or absorbing glass for measuring a high temperature, the transmission of the sector or absorbing glass that must be employed is larger for the longer wave length than for the shorter wave length.—J. S. G. T.

*Viscosimeter; Simple form of —.* H. Mallison. Chem.-Zeit., 1921, 45, 135.

AN apparatus for the rapid determination of comparative viscosities of oils, varnishes, etc., when exact results are not required and the amount of the sample is limited, consists of a set of tubes supported in a frame so that they can be quickly turned from a vertical to a nearly horizontal position. The oil to be examined is placed in one of the tubes, and another control tube is filled with an oil of known viscosity, and after turning the frame to the

nearly horizontal position, the rates of flow of the oil streams are compared.—A. de W.

*Indicator properties of two new phthaleins (1,2,3-xylenolphthalein and ortho- $\alpha$ -naphtholphthalein).* W. Csányi. Z. Elektrochem., 1921, 27, 64–68.

THE phthalein from 1,2,3-xylenol and phthalic anhydride should prove a useful indicator for a range of hydrogen-ion concentration on the alkali side corresponding with that covered by methyl orange on the acid side. The change from colourless to blue is completed in the narrow range between  $pH=8.9$  and  $10.2$ . The colour of the indicator is unaffected by excess of alkali or by alcohol. The phthalein from  $\alpha$ -naphthol, on the other hand, though it would serve as an indicator for approximately the same range of hydrogen-ion concentration, shows a less sharp colour change and is decolorised by excess of alkali. Its slight solubility in water and alcohol also diminishes its value as an indicator.—E. H. R.

*Hydrogen-ion concentration; Determination of — by the colorimetric method and an apparatus for rapid and accurate work.* E. Van Alstine. Soil Sci., 1920, 10, 467–479.

AN apparatus is described for use where a large number of determinations of hydrogen-ion concentration of various solutions are necessary. It is adapted for use with the double-tube standards of Gillespie (Soil Sci., 1920, 9, 115–136; *cf.* J., 1920, 530 A). A graph is given by the aid of which a double-tube colour standard for any  $pH$  value can be easily and quickly prepared.—W. G.

*Sodium and potassium ions; Detection of — in the presence of magnesium ions.* E. Ludwig and H. Spirescu. Bul. Soc. Chim. România, 1920, 2, 77–82.

THE filtrate obtained after the removal of calcium, strontium, and barium by ammonium carbonate or oxalate is evaporated to dryness, and the residue ignited. One portion of the residue is examined for magnesium in the usual way. Another small portion is added to a drop of a solution containing copper acetate, lead acetate, acetic acid, and sodium nitrite on a microscope slide. The appearance of a black precipitate consisting of cubic crystals of the complex nitrite,  $K_2CuPb(NO_3)_6$ , indicates the presence of potassium ions. A third small portion of the residue is added to a drop of a concentrated solution of potassium carbonate on a slide and the liquid is evaporated to dryness. To this residue a drop of a solution of potassium pyroantimonate is added. The appearance of a characteristic crystalline precipitate of sodium pyroantimonate indicates the presence of sodium.—W. G.

*Hydrogen ion concentration at which iron is precipitated from hydrochloric acid solution by ammonia, sodium hydroxide, and hydrogen sulphide.* H. E. Patten and G. H. Mains. J. Assoc. Off. Agric. Chem., 1920, 4, 233–234.

WHEN a dilute solution of iron in hydrochloric acid is treated with ammonia, sodium hydroxide, or hydrogen sulphide, the first formation of ferrous sulphide or of colloidal ferric hydroxide occurs when the hydrogen ion concentration is slightly above  $pH 3.3$ .—W. P. S.

*Iron and manganese; Separation and determination of —.* C. Kollo. Bul. Soc. Chim. România, 1920, 2, 89–95.

FOR the estimation of iron and manganese in the presence of one another the mixture of salts is dissolved in a little water, acidified, and the iron completely oxidised to the ferric state by means of hydrogen peroxide. The iron is then precipitated as ferric hydroxide by the addition of an excess of a 10% solution of hexamethylenetetramine. The

precipitate is filtered off, dried, ignited, and weighed as ferric oxide. The filtrate is evaporated to dryness, and the residue evaporated with a small quantity of strong sulphuric acid, then ignited, and the manganese weighed as manganous sulphate.

—W. G.

*Cations of the group: Ag, Pb, Hg; Detection and identification of the —.* C. Kollo. Bul. Soc. Chim. România, 1920, 2, 95—99.

MERCURIOUS mercury is tested for by the addition of hydrochloric acid to a small portion of the solution, the precipitate being collected on a filter paper and a drop of ammonia added. To the bulk of the solution hydrochloric acid is added, and chlorine is bubbled through until the whole of the mercurous chloride is converted into soluble mercuric chloride. The precipitated lead and silver chlorides are filtered off and separated by dissolving the lead chloride in aqueous sodium hydroxide. In the filtrate the presence of lead is confirmed by the addition of acetic acid and potassium iodide. The presence of silver is confirmed by warming the precipitate left after solution of the lead chloride with a little dextrose and observing the reduction.

—W. G.

*Nickel and cobalt; Qualitative and quantitative determination of —.* M. Matsui and T. Nakazawa. Mem. Coll. Sci., Kyoto, 1920, 4, 265—271.

NICKEL hydroxide reacts directly with a 1% alcoholic solution of dimethylglyoxime. In presence of other hydroxides nickel may be detected by boiling the mixture for a short time with excess of dimethylglyoxime solution, diluting considerably with water, cooling, and shaking with ether. On standing, the nickel dimethylglyoxime floats up with the ether. Cobalt dimethylglyoxime may be detected in solution by adding a drop or two of yellow ammonium sulphide solution, when a very intense red colour is produced if cobalt is present. The test is very delicate if a large excess of dimethylglyoxime is used, but, since ferrous dimethylglyoxime also gives a red colour, iron must not be present. Both nickel and cobalt-dimethylglyoximes can be electrolysed in alkaline solution, and the metal can be thus determined with accuracy. It is possible to separate the two metals in the usual way with dimethylglyoxime and then determine each separately by electrolysis. The solution should contain 0.02—0.05 g. of the metal in 10 c.c. of electrolyte (0.2 g. of dimethylglyoxime and 5.5 g. of caustic soda for Ni or 6.5 g. for Co per 100 c.c.). The electrolysis should take place below 50 mins., using 800 rotations, with a current of 3.5 amps. at 3.8—4.0 volts for nickel and 4.0—4.5 volts for cobalt.—E. H. R.

*Micro-analyses; Weighing of the precipitation vessel with the precipitate in quantitative —.* Two methods based on this principle. E. Gartner. Monatsh., 1920, 41, 477—498.

Two methods are described which permit of the estimation of precipitates weighing 2—15 mg. with sufficient accuracy, the only requisites being a Kuhlmann microchemical balance and such simple apparatus as is easily constructed in the laboratory. In either case the whole of the operations, comprising the weighing of the substance to be analysed, the precipitation, and the weighing of the precipitate, are carried out in a single vessel, this being either an asbestos filter-tube or a pointed centrifuge tube. A tare 1—2 mg. less in weight than the vessel is prepared, the three necessary weighings then requiring only the tare, 1-cg. weight, and the rider. Adhesion of the precipitate to the wall of the vessel causes no error, and both procedures are found to give accurate results.—T. H. P.

*Mercury; Separation of — from other elements by distillation from hydrochloric acid solution.* W. Strecker and K. Conradt. Ber., 1920, 53, 2113—2127.

MERCURIC chloride distils slowly when a current of hydrochloric acid gas is passed through its boiling aqueous solution, but the rate of distillation can be greatly accelerated by addition of concentrated sulphuric acid or phosphoric acid, and increasing the temperature. Complete distillation in  $\frac{1}{2}$  hr. was obtained by passing a current of hydrochloric acid gas through a sulphuric acid solution of mercuric chloride at 160°—170° C., whilst a mixture of concentrated hydrochloric acid with 10% aqueous hydrobromic acid was dropped into the distillation flask. It was subsequently found that in place of the current of hydrochloric acid gas a stream of air or nitrogen can be used, its action being purely mechanical; and, when phosphoric acid was used, complete distillation was obtained in 1 hr. by merely dropping water into the distillation flask at 150°—160° C. The above distillation process was used for the separation of mercury from copper, cadmium, lead, iron, barium, and other metals. In the case of lead it is better to use phosphoric rather than sulphuric acid, since lead sulphate encloses the mercuric chloride and retards the distillation. Separation from bismuth was incomplete, because the chloride of this element is volatile to some extent. (Cf. J.C.S., Jan.)—E. H. R.

*Perchloric acid as a dehydrating agent in the determination of silica.* H. H. Willard and W. E. Cake. J. Amer. Chem. Soc., 1920, 42, 2208—2212.

SILICA can be rendered insoluble by boiling with concentrated perchloric acid solution (60 to 70%) for a short time. Less silica remains in the filtrate than by the usual method of evaporating the hydrochloric acid solution to dryness, and the process is far more rapid. The perchlorates formed are dissolved instantly on dilution with water, leaving pure silica uncontaminated by difficultly soluble salts.—J. R. P.

*Nitric acid; Gravimetric determination of —.* L. W. Winkler. Z. angew. Chem., 1921, 34, 46.

THE following procedure is recommended for the determination of nitric acid as nitron nitrate (Busch, J., 1905, 291). 100 c.c. of the neutral solution, containing 0.01—0.05 g.  $\text{NO}_3$ , is acidified with 1 c.c. of glacial acetic acid, then treated at 60°—70° C. with 10 c.c. of a perfectly clear 10% solution of nitron acetate, and allowed to stand for 24 hrs. in the dark at 15°—20° C. The precipitate is collected on a small pad of cotton wool in a cup-shaped filter, and washed with 50 c.c. of a saturated solution of nitron nitrate at the ordinary temperature. The precipitate is sucked as dry as possible, and dried at 100° C. for 2—3 hrs. Sulphates and iodates are without influence on the results, but chlorides produce small errors, corrections for which are shown in a table.—J. H. L.

*Technical gas analysis, based on the measurement of thermal conductivity.* M. Möller. Wiss. Veröffentl. Siemens Konzern, 1920, 1, 147—153. Chem. Zentr., 1921, 92, II., 210—211.

A METHOD of technical gas analysis, especially for the determination of hydrogen, carbon dioxide, and methane, is based upon the difference in the respective thermal conductivities of certain gases. Carbon monoxide, nitrogen, and oxygen are not differentiated from one another by the method, which is based upon the different cooling effects experienced by two exactly similar heated wires contained respectively within one of two exactly similar very narrow cylindrical metal chambers and surrounded respectively by the gas to be investigated and a standard

gas (*cf.* J., 1920, 470 A, 706 A; 1921, 66 R). Air, or the gas under investigation, freed from the impurities, may be used as standard gas. Convection effects are eliminated as far as possible. The two wires constitute two arms of a Wheatstone network in which a constant electric current is maintained, and the galvanometer scale is calibrated so that the deflection gives directly the percentage of one constituent present. The method is applicable only to gases containing known constituents. With a portable apparatus a determination of hydrogen in air can be made in 2-3 secs. The method can be used for indicating and controlling the combustion in furnaces at a distance.—J. S. G. T.

*Carbon dioxide and oxygen in air and flue gas; Indicators for —.* L. H. Milligan, D. O. Crites, and W. S. Wilson. U.S. Bureau of Mines, Tech. Paper 238, 1920. 21 pages.

APPARATUS has been devised for the testing of air for carbon dioxide and oxygen and of flue gas for carbon dioxide, which is portable, reasonably accurate, and capable of use by any person without chemical training. The first gives an accuracy of  $\pm 0.2\%$   $\text{CO}_2$ , and the latter two of  $\pm 0.5\%$   $\text{CO}$  or  $\text{O}_2$ . The carbon dioxide indicators are on familiar principles, employing a caustic soda solution as absorbent, but incorporate the following points. The gas is saturated with water vapour by drawing it in through moist cheese cloth. The absorption of carbon dioxide during the taking of the sample is prevented by a layer of Russian "white oil" on the caustic soda in the sample chamber. In the calibration of the scale corrections are made for the aqueous vapour absorbed by caustic soda and for the diminution of pressure due to absorption of carbon dioxide. In the case of flue gas a filtering and cooling arrangement for the gas is provided. In the oxygen apparatus alkaline pyrogallate is used as absorbent, and the air is dried and freed from carbon dioxide by passing through a tube filled with soda lime and calcium chloride. Numerous comparative tests with the indicators and with the Haldane and Orsat apparatus are given.—C. I.

See also pages (A) 172, *Lubricating oils* (Lichthardt), 173, *Aniline* (Sabalitschka and Schrader), 174, *Cellulose in bast fibres* (Uyeda), 175, *Kraft paper pulps* (Wilen), 176, *Detergents* (Heermann), 177, *Sulphur dioxide and trioxide* (Kruhl); *Ammonia* (Kollo and Teodossiu); *Iodides and iodates* (Thüringer), 181, *Sulphur in iron and steel* (Cartoceti); *Chromium in steel* (Evans), 183, *Molybdenum* (Bonardi and Barrett), 185, *Gas analysis* (Erlwein and Becker); *Point discharge in nitrogen* (Pirani and Lax), 186, *Turpentine oil* (Salvaterra), 188, *Chrome tanning liquors* (Klaber), 189, *Tanning extracts* (Forbes, also Veitch); *Tannin* (Veitch), 191, *Moisture in sugar etc.* (Spencer); *Catalase content of milk* (Machens and Cordes), 192, *Theobromine in cocoa* (Wadsworth), 193, *Theobromine and caffeine* (Malmy), 196, *Mercury fulminate and sodium thiosulphate* (Dupré and Dupré).

#### PATENTS.

*Admixture of one gas with others; Indication of the —, utilising the change of thermal conductivity occurring in a standard gas by such admixture.* Siemens u. Halske A.-G. G.P. 306,397, 23.4.14.

In a device wherein contamination of one gas with another is ascertained from a comparison of the respective heat losses from a heated wire subjected to the action respectively of the mixture and of a standard gas, the effect of the velocity of the stream of gas upon the indication is allowed for by mounting the heated wire very close to the walls of its containing chamber. The small residual effect of

the velocity of the gas stream can be compensated by causing the standard gas to circulate in a closed space.—J. S. G. T.

*Gaseous mixtures; Process and apparatus for the continuous determination of constituents of — by the method of absorption.* A. Schneider. G.P. 323,975, 3.4.17.

THE gas to be analysed is passed, at constant temperature, into a vessel of constant volume, and when a definite pressure is attained therein a tap is automatically turned so that a definite constant fraction of the gas is transferred to an absorption vessel, where a constituent of the gas mixture is absorbed and its amount determined. The tap is operated by an electrical contact device.

—J. S. G. T.

*Specific gravity; Apparatus for weighing or for ascertaining — [by the displacement method].* H. G. Evans. E.P. 158,018, 15.9.19.

*Specific gravity of flowing liquids etc.* E.P. 158,151. See 1.

#### Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

#### I.—GENERAL; PLANT; MACHINERY.

##### APPLICATIONS.

Baines and Morgan. Centrifugal separators. 5862. Feb. 21. (Australia, 19.2.20.)

Brown and Coldrey. Drying apparatus. 6363. Mar. 2.

Calvert. Gaseous reactions. 6312. Feb. 24.

Chemical Fuel Co. of America. Distillation processes. 7025. Mar. 3. (U.S., 9.3.20.)

Chemical Fuel Co. of America. Processes of catalysis. 7026. Mar. 3. (U.S., 18.3.20.)

Crawford and Kelly. Filters. 6185. Feb. 24.

Dunlop Rubber Co. (Lewis and Green). Apparatus for drying materials carrying a volatile inflammable solvent and for recovering the solvent. 7115. Mar. 4.

Gardner. Mixers and driers. 6746. Mar. 1.

Gill and others. 6418. See 11.

Gill (Sharples Speciality Co.). Processes for resolving emulsions. 6959. Mar. 3.

Graham and Honeywood. Crushing or grinding machinery. 5851. Feb. 21.

Grainger and Shadbolt. Apparatus for drying solid chemicals. 6679. Feb. 28.

Heenan and Froude, and Walker. Separation of dust etc. from air or gases. 7166. Mar. 5.

Humphries. Packing etc. material for scrubbing and absorption towers, distilling columns, etc. 6521. Feb. 26.

Kennedy. Mixing and agitating machines. 6319. Mar. 2.

Kennedy. Gyrotory crushing apparatus. 6986-8. Mar. 3. (U.S., 23.10.17.)

Lumpp. Centrifugal drying machines. 7027. Mar. 3. (Fr., 3.3.20.)

Lunt. Method of drying colloids. 6018. Feb. 22. (U.S., 7.4.16.)

Righty. Drying materials containing water etc. 6343. Feb. 25.

Soc. Gén. d'Evaporation. Crystallisation. 5890. Feb. 21. (Fr., 5.3.20.)

Soc. Gén. d'Evaporation. 5839. See X.

Tulloch. Recovery of salts from their solutions. 6026. Feb. 22.



Wade (Grigor). Filter presses. 7094. Mar. 4.  
Withers (Penning). Centrifugal separating apparatus. 6794. Mar. 1.

COMPLETE SPECIFICATIONS ACCEPTED.

24,766 (1919). Conder and Vivian. Crushing-mills of the roller and ring type. (159,244.) Mar. 9.  
29,036 (1919). Phillips and Blaikie. Furnaces. (159,298.) Mar. 9.  
29,781 (1919). Melton and Downs. See XII.  
30,177 (1919). Maass. Separating aqueous and other vapours from liquids and solids and preparing dilute sulphuric acid. (159,054.) Mar. 2.  
980, 1112, and 1115 (1920). Niessen. See XIX.  
5459 (1920). Schjelderup. Drying material containing liquid. (139,478.) Mar. 9.  
19,516 (1920). Tixier. Filters. (147,582.) Mar. 2.  
20,633 (1920). Deutsche Maschinenfabr. A.-G. Apparatus for separating solid substances from gases and vapours, especially blast-furnace gases. (148,802.) Mar. 9.  
24,873 (1920). Klingenberg. Filter for separating impurities from gas or steam. (150,694.) Mar. 9.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Buell. Burning pulverised fuel. 6356. Feb. 25. (Australia, 23.4.20.)  
Christenson and Hedman. 6313, 6412, 6446. See VII.  
Gill, Morton, and South Metropolitan Gas Co. Method of making furnaces, coke ovens, retort settings, etc. 6418. Feb. 25.  
Gill (Sharples Speciality Co.). 6959. See I.  
Harvie, and Harvie and Co. Wet-coal vertical drier. 6486. Feb. 26.  
Johnson, and M.-L. Experimental, Ltd. Producing and utilising smoke-producing compositions. 5877. Feb. 21.  
Jones, and Minerals Separation, Ltd. Concentration of coal. 5967. Feb. 22.  
Kansas City Gasoline Co. Art of cracking hydrocarbons. 6014-5. Feb. 22. (U.S., 12.3 and 23.4.20.)  
Lewis. Sorting portions of coal according to content in earthy or mineral matter. 6903. Mar. 2.  
Lulofs, and Powdered Fuel Plant Co. Means for drying coal etc. 6114. Feb. 23.  
Marks (Soc. des Fours à Coke Semet-Solvay et Piette). Coke ovens. 6760. Mar. 1.  
Pierson and Pierson. Generators for making low-grade gas. 6286. Feb. 24.  
Plauson and Vielle. Manufacture of hydrocarbons from carbonaceous substances etc. 7160. Mar. 5.  
Reid. Production of gas and carbonaceous materials. 6010. Feb. 22.  
Sangunetti. Coloured artificial asphaltum. 7008. Mar. 3.  
Seigle. Distilling and gasifying peat etc. 6273. Feb. 24.  
Seigle. Transforming and distilling hydrocarbons. 6274. Feb. 24.  
Smith and Tulloch. Gas-producers. 7139. Mar. 4.  
Szarvasy. Manufacture of pure retort carbon. 6645. Feb. 28. (Hungary, 29.1.18.)  
Torfverwertungsges. Dry distillation and coking of raw peat etc. 6004. Feb. 22. (Ger., 23.2.20.)  
Tulloch. Fuel. 6023. Feb. 22.  
Tulloch. Fuel for gas-producers. 6532. Feb. 26.

COMPLETE SPECIFICATIONS ACCEPTED.

18,026 (1918). Barbet et Fils et Cie. Removing tar from products of distillation of wood, coal, etc. (158,915.) Mar. 2.

18,163 (1919). Wade (Standard Oil Co.). Manufacture of fuel from crude petroleum oil or residues. (158,918.) Mar. 2.  
20,152 (1919). Hood, Spence, and Spence and Sons. Lubricants. (158,922.) Mar. 2.  
25,655 (1919). Gercke. Distilling bituminous coal, brown coal, and shale by superheated steam. (159,246.) Mar. 9.  
23,832 (1919). Edser, Sulman, and Jones. Treatment of materials containing coal. (159,285.) Mar. 9.  
29,490 (1919). Fleming. Converting hydrocarbons into hydrocarbons of lower boiling-point. (135,854.) Mar. 2.  
2315 (1920). Duckham. Manufacture of mixtures of pulverised fuel with tar or other liquid fuel. (159,089.) Mar. 2.  
5099 (1920). Tully. Gas-producers. (159,409.) Mar. 9.

III.—TAR AND TAR PRODUCTS.

APPLICATION.

Soc. Anon. des Matières Colorantes et Prod. Chim. de St.-Denis, and Wohl. Chlorotoluenes. 7014. Mar. 3. (Fr., 6.3.20.)

COMPLETE SPECIFICATION ACCEPTED.

18,026 (1918). Barbet et Fils et Cie. See II.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

Atack and Soutar. Manufacture of anthraquinone dyestuffs. 6063. Feb. 23.  
Drey, and Williams and Co. Production and utilisation of colouring matters. 6192. Feb. 24.  
Imray (Soc. Chem. Ind. Basle). Manufacture of mordant dyestuffs and chromium compounds thereof. 6036. Feb. 22.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

British Cellulose and Chem. Manuf. Co., and Mallock. Apparatus for spinning artificial threads etc. 6969. Mar. 2.  
Cross (Technochemia A.-G.). Preliminary treatment of cellulose for the manufacture of artificial silk. 7135. Mar. 4.  
Du Pont de Nemours and Co. Cellulose ester compositions. 6897. Mar. 2. (U.S., 19.6.20.)  
Rütgerswerke A.-G., and Teichmann. Dissolution of wood etc. 7077. Mar. 4. (Ger., 17.3.20.)  
Steinhilber. Manufacture of paper-pulp. 6972. Mar. 3.  
Trostel. Improving hair, wool, and furs. 6515. Feb. 26.  
Wade (International Paper Co.). Manufacture of paper. 7132. Mar. 4.

COMPLETE SPECIFICATIONS ACCEPTED.

23,190 (1919) and 23,982-3 (1920). Glanzfäden A.-G. Producing a spun material resembling wool or cotton from viscose solutions. (135,205, 152,349, 152,350.) Mar. 2.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Brandwood and Brandwood. Dyeing yarns. 6722. Mar. 1.  
Copley. Mercerising yarns in hank form. 6353. Feb. 25.



Drey, and Williams and Co. 6192. *See* IV.  
Renck. Production of gelatin printing plates.  
6697. Mar. 1. (Ger., 2.9.20.)

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

### APPLICATIONS.

Christenson and Hedman. Production of ammonium chloride in coking or distilling coal. 6313. Feb. 24. (Sweden, 3.3.20.)  
Christenson and Hedman. Production of ammonium chloride in distilling etc. alum shale etc. 6412. Feb. 25. (Sweden, 27.3.20.)  
Christenson and Hedman. Production of ammonium chloride. 6446. Feb. 25. (Sweden, 6.10.20.)  
Collins. 7200. *See* XI.  
Eustis. Recovering sulphur dioxide from furnace gases etc. 6783-4. Mar. 1.  
Hansford. Apparatus for drying ammonium sulphate etc. 6686. Mar. 1.  
Hultman. Manufacture of chrome alums. 6122. Feb. 23. (Sweden, 1.3.20.)  
Kelly and Walker. Manufacture of borax and boric acid. 7045. Mar. 3.  
Kilburn. 6618. *See* XIII.  
Norsk Hydro-Elektrisk Kvaestofaktieselskab. Manufacture of ammonia. 6801. Mar. 1. (Norway, 9.3.20.)  
Potter and Robinson. Manufacture of chromium compounds. 6690. Mar. 1.  
Stohr. Manufacture of ferruginous preparations. 6786. Mar. 1. (Austria, 9.3.20.)  
Tulloch. 6026. *See* I.

### COMPLETE SPECIFICATIONS ACCEPTED.

23,318 (1919). Evans and Evans. *See* X.  
28,355 (1919). Kelly and Jones. Preparation of alkali pentaborates direct from boron ores. (158,992.) Mar. 2.  
29,303 (1919). New Jersey Zinc Co. Production of metal oxides and other compounds of metals. (147,530.) Mar. 9.  
30,075 (1919). Pascal. Manufacture of liquid sulphurous acid from dilute sulphurous acid gas. (159,337.) Mar. 9.  
30,090 (1919). General Chemical Co. Production of oxides of nitrogen. (136,158.) Mar. 9.  
30,177 (1919). Maass. *See* I.  
1712 (1920). Sieurin. Production of aluminium oxide from aluminium chloride. (159,086.) Mar. 2.

## VIII.—GLASS; CERAMICS.

### APPLICATIONS.

Hailwood. Glass manufacture. 7149. Mar. 5.  
Pilkington, and Pilkington Bros. Apparatus for gathering glass from molten mass. 6984. Mar. 3.  
Rashleigh. Means for drying clay etc. 6806. Mar. 1.

### COMPLETE SPECIFICATION ACCEPTED.

14,371 (1920). Danner. Forming sheet glass. (159,114.) Mar. 2.

## IX.—BUILDING MATERIALS.

### APPLICATIONS.

Dagnall. Heat-insulation or fireproof bricks, tiles, etc. 6563. Feb. 23.  
Laube. Wood-preserving method. 6399. Feb. 25. (Ger., 28.2.20.)  
Pearson. Manufacture of material for road etc. surfaces. 7141. Mar. 4.

Sanguinetti. 7008. *See* II.  
Strehler. Manufacture of artificial stone. 6673. Feb. 28.

### COMPLETE SPECIFICATIONS ACCEPTED.

22,588 (1919). Hughes, Brown, and Hill-Jones, Ltd. Manufacture of bricks, slabs, tiles, etc. (159,239.) Mar. 9.  
6057 (1920). McLay. Heat non-conducting compositions. (159,411.) Mar. 9.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

### APPLICATIONS.

Angel. Treatment and reduction of sulphide refractory etc. ores. 6232. Feb. 24.  
Basset. Manufacture and refining of iron and steel. 6277. Feb. 24. (Fr., 24.2.20.)  
Eustis. 6783-4. *See* VII.  
Hardy and Stephenson. Siemens etc. furnaces. 7041. Mar. 3.  
Morgan Crucible Co., and Speirs. Electrically heated melting etc. furnaces. 6912. Mar. 2.  
Passalacqua. Soldering aluminium. 6412. Feb. 25. (Fr., 23.2.20.)  
Perkins. Treatment of oxidised ores. 5899. Feb. 21.  
Platt. Metallurgical etc. furnaces. 6291. Feb. 24.  
Soc. Gén. d'Evaporation. Apparatus for leaching minerals. 5889. Feb. 21. (Fr., 30.3.20.)  
White (American Smelting and Refining Co.). Treatment of tin. 6299. Feb. 24.

### COMPLETE SPECIFICATIONS ACCEPTED.

2690 (1914). Zavelberg. Roasting-furnaces. Mar. 9.  
23,318 (1919). Evans and Evans. Recovery of tin and zinc chloride from tin pot skimmings. (158,926.) Mar. 2.  
28,546 (1919) and 6733 (1920). Hurst and Ball. Aluminium alloys. (159,008.) Mar. 2.  
28,763 and 30,472 (1919). Ballantine. Production of ferro-alloys. (159,280.) Mar. 9.  
28,999 (1919). Taylor and Partington. Treatment of ores etc. by flotation. (159,025.) Mar. 2.  
30,951 (1919). Collins. Winning of tin. (159,071.) Mar. 2.  
31,814 (1919). Støren and Johanson. Treating magnetic or weakly magnetic iron ore containing magnetic ferrous sulphide. (159,380.) Mar. 9.  
6912 (1920). Metallind. Schiele u. Bruchsalser. Increasing resistance of aluminium to acid and alkaline liquids. (140,069.) Mar. 9.  
8213 (1920). British Thomson-Houston Co. (General Electric Co.). Surface treatment of metals. (159,102.) Mar. 2.  
14,794 (1920). Tyrrell. Briquetting ores. (144,276.) Mar. 2.  
20,633 (1920). Deuts. Maschinenfabrik. *See* I.

## XI.—ELECTRO-CHEMISTRY.

### APPLICATIONS.

Chloride Electrical Storage Co., and Heap. Storage batteries or accumulators. 6137-8. Feb. 23.  
Chloride Electrical Storage Co. (Ford). Storage batteries or accumulators. 6655 and 6759. Feb. 23 and Mar. 1.  
Collins. Electrolysis of brine. 7200. Mar. 5.  
Imbery. Electric furnaces. 6595. Feb. 23.  
Morgan Crucible Co., and Spiers. 6912. *See* X.  
Reid. Electric furnaces. 6011. Feb. 22.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 3583 (1917). Schuster. Galvanic cells. (158,911.) Mar. 2.  
 28,154 (1919). Mond (Internat. Precipitation Co.). Apparatus for electrical treatment of gases. (158,982.) Mar. 2.  
 7944 (1920). British Thomson-Houston Co. (General Electric Co.). Compositions for insulating and protecting electrical apparatus etc. (159,421.) Mar. 9.

## XII.—FATS; OILS; WAXES.

## APPLICATIONS.

- Douglas and Sons, and Nicol. Treatment of edible fats. 6787. Mar. 1.  
 Gill (Sharples Speciality Co.). 6959. *See* I.  
 Henkel u. Co. Treatment of liquid soaps etc. 6670. Feb. 28. (Ger., 16.12.20.)  
 Kayser. Refining vegetable oils. 6648. Feb. 28.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 23,585 (1919). Erslev. Cooling or allowing to crystallise concentrated fatty emulsions. (134,815.) Mar. 9.  
 29,731 (1919). Melton and Downs. Recovering and utilising solvent from the air from apparatus for extracting oil, fat, etc. (159,039.) Mar. 2.  
 599 (1920). Townsend. Soap. (159,083.) Mar. 2.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

## APPLICATION.

- Kilburn (Titanium Pigment Co.). Manufacture of composite titanic oxide products. 6618. Feb. 28.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

## APPLICATIONS.

- Bilbrough. Manufacture of pure or filled rubber from latex. 6737. Mar. 1.  
 Davidson. Treatment of latex. 6041. Feb. 22.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 28,593 (1919). Perkin, Mandleberg, and Mandleberg and Co. Compositions containing india-rubber. (159,014.) Mar. 2.  
 10,829 (1920). Davidson. Treatment of raw rubber when freshly coagulated from latex. (159,106.) Mar. 2.

## XV.—LEATHER; BONE; HORN; GLUE.

## APPLICATIONS.

- Beretta. Tanning arrangement for hides etc. 5860. Feb. 21. (Ital., 20.2.20.)  
 Carmichael and Ockleston. Tanning. 6221. Feb. 24.  
 Hell. Tanning hides etc. 6798. Mar. 1.  
 Renck. 6697. *See* VI.

## XVI.—SOILS; FERTILISERS.

## APPLICATIONS.

- Desmond and Tisdall. Fertiliser. 6765. Mar. 1.  
 Lo Monaco. Chemical fertilisers. 6434. Feb. 25. (Ital., 26.2.20.)

## XVIII.—FERMENTATION INDUSTRIES.

## APPLICATIONS.

- Crawford. Fermentation of alcoholic beverages. 6700. Mar. 1.  
 Gilmour. Manufacture of yeast. 6107. Feb. 23.  
 Jensen (Corby). Making, treating, and preparing yeast. 6636. Feb. 28.

- Kashiwaga. Preparation of diastase or a solution of diastase. 6235. Feb. 24.

## XIX.—FOODS; WATER PURIFICATION; SANITATION.

## APPLICATIONS.

- Apsley. Apparatus for treating sewage etc. 7076. Mar. 4.  
 Boldy and Waite. Machines for separating solid matter from trade effluent etc. 6474. Feb. 26.  
 Brunet, and Soc. Anon. Maison Detilleux. Disinfecting or sterilising. 7222. Mar. 5.  
 Carpmael (Bayer u. Co.). Disinfecting, insecticidal, and fungicidal compositions. 6419. Feb. 25.  
 Corn Products Refining Co. Separating gluten from starch. 7024. Mar. 3. (U.S., 8.3.20.)  
 Dorr Co. Treatment of sewage. 7219. Mar. 5. (U.S., 27.3.20.)  
 Douglas and Sons, and Nicol. 6787. *See* XII.  
 Raimbert. Sand filters. 5883. Feb. 21. (Fr., 23.3.20.)  
 Spear and Spear. Manufacture of food products. 5879. Feb. 21.  
 Wanklyn. Treatment of germ of cereals. 6830. Mar. 2.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 16,189 (1919). Greville. Treatment of flour. 158,917. Mar. 2.  
 29,245 (1919). Nicholson. Extraction of juices, pectin, etc. from fruit etc. (159,311.) Mar. 9.  
 30,978 (1919). Townsend. Milk food. (159,362.) Mar. 9.  
 990, 1112, and 1115 (1920). Niessen. Boiling and drying organic substances, particularly offal. (137,828, 137,841, 137,844.) Mar. 2 and 9.  
 18,852 (1920). Sykes. Apparatus for dehydrating food products. (146,917.) Mar. 2.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

## APPLICATIONS.

- Carpmael (Bayer u. Co.). Manufacture of ethyl-one derivatives. 6160. Feb. 23.  
 Elektro-Osmose A.-G. Manufacture of swine-fever serum. 6531. Feb. 26. (Ger., 27.2.20.)  
 Soc. Chim. Usines du Rhône. Manufacture of oxyaldehydes and their derivatives. 6289, 6450. Feb. 24 and 25.  
 Szarvasy. Manufacture of methyl alcohol from methyl chloride. 7142. Mar. 4. (Hungary, 16.7.14.)

## COMPLETE SPECIFICATIONS ACCEPTED.

- 6209 (1920). Datta. Treatment of waste from growing tea plants and obtaining caffeine. (159,097.) Mar. 2.  
 13,349 (1920). Lilienfeld. Manufacture of dialkyl sulphates. (143,260.) Mar. 9.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## APPLICATIONS.

- Donisthorpe. Colour photography. 6859. Mar. 2.  
 Luboshey. X-ray photography. 6901-2. Mar. 2.

## XXIII.—ANALYSIS.

## APPLICATION.

- König. Measuring density of gases. 7162. Mar. 5. (Ger., 5.3.20.)

## COMPLETE SPECIFICATIONS ACCEPTED.

- 30,637 (1920). Siemens u. Halske A.-G. Photometers. (153,323.) Mar. 2.

**I.—GENERAL; PLANT; MACHINERY.**

*Anemometer.* Gerdién and Holm. See XXIII.

**PATENTS.**

*Combustion furnace.* W. L. Harder, Assr. to Harder Furnace and Engineering Corp. U.S.P. 1,354,741, 5.10.20. Appl., 13.3.19.

POWDERED fuel is injected into a furnace between streams of heated air which are independently regulated so that combustion takes place at any desired point in the furnace.—W. F. F.

*Surface-condensers; Process of keeping — free from scale and sludge.* Maschinenbau-A.-G. Balcke. E.P. 135,189, 10.11.19. Conv., 19.2.18.

A PREDETERMINED quantity of an acid such as hydrochloric, acetic, or formic acid, is added to the cooling water to convert carbonates into easily soluble salts; the process is applicable to condensers (but not to boilers) because the temperatures used are insufficient to cause dissociation of the chlorides or other salts formed. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 2798 of 1874 and 139 of 1911.)—B. M. V.

*Evacuating [dangerous] fluid from containers; Method of and apparatus for —.* F. N. Pickett, W. J. Palmer, and G. H. Tatham. E.P. 158,333, 30.10.19.

THE container, e.g., a shell containing "mustard gas," is opened by a punch which drives in the soft iron sealing plug, and evacuated by a pipe connected with a source of vacuum. To prevent escape of gas during the operations the shell is inserted through an opening provided with a rubber airtight collar into a chamber in which also a vacuum is maintained, means being provided for operating the punch and emptying pipe without destroying the vacuum. The shell may be held in place by means of an electro-magnet.—B. M. V.

*Filters.* J. Miller, and G. Fletcher and Co., Ltd. E.P. 158,387, 7.11.19.

A FILTER which may be used, e.g., for filtering sugar stock through megass, comprises a chamber mounted on hollow trunnions, through which the liquor to be filtered is admitted and exhausted. The chamber is tilted to receive a charge of filtering medium, then placed in a vertical position, and a lid and pressure plate, both supported by an overhead yoke, are lowered respectively upon and into the filter chamber by means of a screw; the pressure plate continues to move downward after the lid has come to rest until the filtering medium is sufficiently compacted. The liquor to be filtered is then pumped through, and should the filtrate not be clear, additional pressure may be applied to the plate without stopping the operation of filtering.—B. M. V.

*Filtering apparatus; Arrangement and construction of —.* C. J. Haines. E.P. 158,663, 7.11.19.

FILTERING frames, suitable for use in the apparatus described in E.P. 6272 of 1900 and 27,007 of 1903, are constructed of two perforated sheets kept apart by a corrugated sheet and held in place by a cast base and frame of channel section. The perforated sheets may be covered by wire gauze and that in turn by the filtering medium.—B. M. V.

*Filter.* J. P. Probst. U.S.P. (A) 1,367,324 and (B) 1,367,325, 1.2.21. Appl., 14.2.19 and 12.6.19.

(A) A VERTICAL casing is provided near the top and the bottom with horizontal ridges projecting

inwards, each ridge supporting an annular flange. Removable interchangeable supports rest on the flanges and a cylindrical filter screen is mounted on one of the supports. Filtering material is packed in the casing between the bottom of the filter screen and the other support. (B) In a filter of the kind described in (A) liquid passes into an annular space surrounding the casing and thence into a filtering chamber filled with compressed sponges. The liquid then passes into a second chamber within the casing filled with layers of crushed cloth, the second chamber being detachable from the casing. The liquid is discharged through a perforated wall into a second annular space surrounding the second filtering chamber.—W. F. F.

*Filter; Metallurgical and chemical —.* H. B. Faber. U.S.P. 1,363,618, 15.2.21. Appl., 3.4.15.

WITHIN an airtight container is a rotary filter in sections, each section having an independent discharge pipe. Means are provided for supplying pressure medium to the container, for supplying cleansing fluid to the solids collected on the filter, and for providing a counter-pressure in excess of the pressure so as to lift all solids from successive portions of the filter during continuous operation without relieving pressure on the filtering portions. By means of a distributing valve the filtrates from the different discharge pipes can be distributed to different receptacles prior to and subsequent to the application of the cleansing fluid.—J. W. D.

*Filter-press.* M. Neide. G.P. 328,941, 18.4.18.

A FILTER-PRESS is constituted of a number of alternate filter plates and filter frames in juxtaposition, the whole being disposed within a casing provided with an opening through which the filter frames or plates may be withdrawn or replaced either as a whole or in sections.—J. S. G. T.

*Membrane or ultra filters with pores of a definite size; Manufacture of —. Membrane filters, and process of making them.* R. Zsigmondy and W. Bachmann. G.P. (A) 329,060, 5.9.16, and (B) 329,117, 22.8.16.

(A) A SOLUTION of a cellulose ester is poured upon plates, or soaked into the pores of textile fabrics or similar material, and is dried in an atmosphere, the relative humidity of which is maintained constant to within a few per cent., the temperature being similarly maintained constant to within a few degrees. Water may be added to the ester before drying, if necessary. A compact filter of small porosity is obtained when the relative humidity employed is low. Employing a higher degree of humidity, the permeability of the resulting filter is increased. The porosity may also be varied by altering the composition of the solvent in which the cellulose ester is dissolved, without altering the concentration of the solution. (B) Membrane filters composed of a cellulose ester and possessing a structure especially adapted for filtration purposes are prepared by using a solvent composed of a mixture of substances in part easily vaporisable and in part vaporisable with difficulty, and submitting the membrane to the action of water and steam.—J. S. G. T.

*Metallic filter gauze; Manufacture of —.* J. Rathjen. G.P. 329,061, 3.9.18.

A METAL gauze of iron, steel, brass, bronze, copper, etc. is coated with another metal by spraying, in such a way that it retains its permeability. If necessary, the gauze may be coated, either before or after the spraying process, with a metal resistant to acids and alkalis, this metal being deposited electrolytically or otherwise. The gauze may be

employed in filter-presses etc. in place of filter cloth, over which it possesses a number of advantages.—J. S. G. T.

*Non-flammable volatile liquid.* A. Henning. E.P. 158,494, 15.7.20.

ETHYL chloride, with or without methyl chloride, is mixed with methyl bromide and, if desired, colouring or perfuming agents, forming a mixture that will vaporise practically as a whole, and will be useful for refrigerating, as a solvent, and for fire extinguishing.—B. M. V.

*Drying apparatus.* C. S. Bedford and A. Oldroyd. E.P. 158,734, 20.11.19.

HOT gases (e.g., flue gases from boilers) are passed in two streams through both compartments of a two-storey drying-chamber, through which the material to be dried is conveyed first in one compartment in the same direction as the gases, returning through the other compartment in the opposite direction to the gases.—B. M. V.

*Cooking thick masses; Apparatus for —.* E. A. Strohmaier and B. A. Hook. U.S.P. 1,366,431, 25.1.21. Appl., 20.12.17.

A COOKING vessel is provided with a rounded bottom and a steam-heating coil near the bottom. A steam pipe extends vertically downwards into the vessel, and is provided with an ejector nozzle, which discharges a jet against the curved bottom so as to circulate the material around the heating coil. —W. F. F.

*Rectifying columns; Apparatus for facilitating the reflux of condensed liquid in —.* C. A. Hartung. G.P. 323,412, 26.6.17.

COLLECTING vessels are arranged below the reflux tubes, and one or more holes are bored in the lower parts of these, affording a restricted outlet for liquid. The holes are of such size that the vessels remain filled under all circumstances.—J. S. G. T.

*Distilling column.* J. Schneible. U.S.P. 1,366,956, 1.2.21. Appl., 2.8.19.

A DISTILLING column consists of a series of annular sections containing chambers, each chamber having a lower annular baffle plate, and an upper circular baffle plate of smaller diameter carrying a series of depending guide vanes which extend to the lower plate.—W. F. F.

*Condenser. Heat exchanger.* J. Schneible. U.S.P. 1,366,957—8, 1.2.21. Appl., 2.8.19.

A CONDENSER comprises a series of superposed horizontal cooling coils in a casing, each coil having an annular baffle plate with a large central opening below it, and a baffle plate above it of larger diameter than the diameter of the opening in the plate below. When used as a heat exchanger, the baffle plate above the coil is of larger diameter, and the annular portion lying above the annular plate below the coil is perforated.—W. F. F.

*Volatile solvents; Process for the recovery of —.* E. Bindschedler. U.S.P. 1,367,009, 1.2.21. Appl., 18.6.20.

GAS containing solvent vapour is brought into contact first with phenols and then with concentrated sulphuric acid to absorb the solvent vapour, which is subsequently recovered from the absorbents. —W. F. F.

*Volatile solvents; Process for the recovery of —.* E. Bindschedler. U.S.P. 1,368,601, 15.2.21. Appl., 23.7.20.

GASES containing a volatile solvent are brought successively into intimate contact with a phenolic

liquid to remove the volatile solvent, with an alkaline solution to remove any phenol carried over, and with concentrated sulphuric acid to remove any remaining solvent.—B. M. V.

*Separation of organic gases or vapours from admixture with air or other gases; Process for —.* Farbenfabr. vorm. F. Bayer und Co. G.P. 310,092, 4.11.16.

THE gaseous mixture is subjected to the action of extremely porous charcoal, prepared in accordance with G.P. 290,656 (F.P. 471,295; J., 1915, 216), which absorbs 5% of its weight of methane, acetylene, or cyanogen, and 50—70% of its weight of ether, methyl or ethyl alcohol, acetone, benzene, ethyl acetate, or acetic acid. The absorbed gases or vapours are subsequently expelled by heating the charcoal in steam.—J. S. G. T.

*Lining metal tanks with hard rubber; Process of —.* E. S. Boyer, Assr. to American Hard Rubber Co. U.S.P. 1,367,231, 1.2.21. Appl., 13.11.20.

THE inner surface of the tank is covered with plastic soft rubber and then with plastic hard rubber, and both layers are vulcanised together and to the tank by the application of steam under pressure. Water under pressure is then introduced into the tank to ensure adhesion during cooling. —W. F. F.

*Osmotic diaphragm.* E. A. Sperry, Assr. to Anaconda Lead Products Co. U.S.P. 1,368,227, 8.2.21. Appl., 26.12.18.

A porous diaphragm for use in a bi-fluid cell is composed of cloth parchmentised to a predetermined extent.—J. S. G. T.

*Liquid, pulverulent, or granular substances; Process by which two or more — are simultaneously atomised and mixed or enabled to interact.* G. A. Krause. G.P. 302,421, 1.8.16.

THE materials are delivered together on to a rapidly rotating disc within a chamber, and the powdery product is either collected in the chamber or withdrawn therefrom by suction. The process may be used for the production of soap powder, or of tin oxide from stannate liquors.—J. H. L.

*Contact masses containing iron, cobalt, nickel, etc. or mixtures thereof, or the oxides; Revivification of —.* Badische Anilin und Soda Fabrik. G.P. 304,341, 9.4.14.

THE contact mass is treated with an aqueous oxidising agent, under such conditions that little, if any, of the contact metal or of the oxide is dissolved. Oxidising acids such as nitric acid, and oxidising salts such as nitrates, chromates, etc., may be employed.—J. S. G. T.

*Washing and cooling gases; Multiple-stage disintegrator for —.* F. Trappmann. G.P. 327,047, 9.1.15.

THE gas and water flow alternately in the same and in opposite directions through a multiple-stage disintegrator constituted of two concentric atomising drums rotating in opposite directions within a chamber. The diameters of the drums increase gradually or discontinuously from the inlet to the outlet end of the chamber. The chamber is divided into compartments by a number of transverse circular plates rotating with the atomising drums. The gas passes inwards through the drums towards the axis in one compartment and outwards in the next and so on, and is submitted to an atomising and centrifugal force of progressively increasing magnitude, owing to the increase in the circumferential velocity of the drums in successive compartments. The quantity of water injected into

the various compartments diminishes in proportion as the diameter of the atomising drum therein increases.—J. S. G. T.

*Reaction column [for absorption, distillation, rectification, etc.].* Soc. J. Bouchayer et Paris et Cie. G.P. 327,164, 2.4.19.

In a plate column the tubes for the gas or vapour are arranged at the corners of a regular hexagon and the tubes for the liquid at the centres of the hexagons. The tubes for the liquid are each surrounded by a sleeve having narrow vertical slits in the portion between adjacent plates and also broader openings at the bottom. The spaces between successive plates are packed with a filling material up to the level of the upper ends of the vertical slits. Liquid which is carried up mechanically through the filling material by the gas or vapour circulates through the slits into the space enclosed by the sleeve and then out through the broader openings at the bottom, whereby the same portion of liquid comes a number of times into contact with the gas or vapour. The column is intended for use in gas washing, for recovery of ammonia from coke-oven and blast-furnace gas, and for the recovery of vapours of volatile liquids etc.—J. S. G. T.

*Electrical purification of gases; Removal of dust precipitated in the —.* H. Zschocke. G.P. 329,062, 28.10.19.

Two concentric tubes, the outer of which is stationary, are employed. To the inner tube, which serves as the electrode on which the dust is precipitated, an oscillatory or jerky motion in the direction of its axis is imparted, whereby the dust precipitated is shaken down into a funnel-shaped receptacle.—J. S. G. T.

*Gas washers, reaction columns, and the like; Sprinkling device for use in —.* C. Still. G.P. 329,118, 3.8.19.

The washing liquid is distributed by a sprayer or similar device, the pipe for supplying liquid thereto being connected, by means of a branch pipe, with the gas chamber at some distance from the point of mission of the spray. Thereby, owing to suction or injector action, the washing fluid is admixed with gas prior to entering the sprinkler, whereby a much finer spray is obtained.—J. S. G. T.

*Separation of solid impurities from gases, vapours, and liquids; Cyclone device for —.* R. Wussow and E. Schierholz. G.P. 329,779, 19.8.19.

The fluid enters and leaves the cyclone tangentially in the direction of rotation of the cyclone about its vertical axis. The cyclone is surrounded by an annular collecting chamber, and the lighter constituents are deposited in a central settling chamber.—J. S. G. T.

*Fluids of low boiling point, with the exception of hydrocarbons [e.g., sulphur dioxide and ammonia]; Process for the recovery of — [from gaseous mixtures].* Apparate-Vertriebs-Ges. m.b.H. G. P. 329,412, 10.5.17.

The gaseous mixture is compressed, then expanded and simultaneously brought into contact with a washing agent. The process is applicable to the recovery of certain constituents, such as sulphur dioxide or ammonia, from gases. In these two cases, the condensed liquid separated by compression, constituted in the one case of a solution of sulphur dioxide and in the other of a solution of ammonia in water, is employed as washing medium, and is sprayed into the expansion chamber. The efficiency of the process is increased by the use of impact or reflecting surfaces to ensure intimate contact between the washing medium and the mist formed during the expansion. In like manner the

cooled gas may be passed into a centrifugal washer into which the washing fluid is sprayed.—J. S. G. T.

*Separation of bubbles and drops of liquid from the vapours rising from boiling liquids; Process for the —.* B. Graemiger. G.P. 329,719, 31.7.19.

The vapour rising from the boiling liquid is made to percolate through a vaporous mist moving with much higher velocity, whereby drops of liquid in the vapour are precipitated.—J. S. G. T.

*Crushing ores and other material; Apparatus for —.* H. W. Hardinge. U.S.P. 1,367,777, 8.2.21. Appl., 6.2.19.

SEE E.P. 148,637 of 1919; J., 1920, 619 A.

*Recuperators for use in connection with furnaces.*

H. Hecker, and Bender und Främb G.m.b.H. E.P. 133,045, 24.9.19. Conv., 2.5.18.

*Cooling apparatus.* G.P. 329,479. See VII.

*Leuchting.* E.P. 158,320. See X.

## IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal; Assay of — for carbonisation purposes.* T. Gray and J. G. King. Tech. Paper No. 1, Fuel Research Board, 1921, 13pp.

THE coal sample is ground to pass through a 60-mesh sieve, and is dried at 105°–110° C. The retort consists of a horizontal hard glass or silica tube 30 cm. long and 2 cm. diam., and is fitted with a side tube leading into a U-tube which acts as a condenser for oil and water. This is connected with a second tube charged with glass beads drenched with sulphuric acid for the absorption of ammonia. The gases are collected in a gas-holder fitted with a constant pressure device. About 20 g. of the coal is placed in the tube, and after all connexions have been made the furnace, previously heated to 300° C., is pushed into position round the retort. As soon as the evolution of the gas occluded by the coal and of the expanded air stops, the temperature of the furnace is gradually raised so that the final temperature, 550° C. to 600° C., is reached in 1 hr. Observations are made of the temperatures at which water and oil appear. Heating at the final temperature is continued for 1 hr. Weighings are made of the coke and oils, and the liquor and gas volumes are measured. The method has been found to give concordant results.—W. P.

*Lignite and lignite-coal mixtures; Carbonisation of —.* A. Viehoff and E. Czako. J. Gasbeleucht., 1920, 63, 379–383.

RESULTS are given of the experimental carbonisation of four coals, two of which were lignites whilst two were typical brown coals. Details are given of the composition of the coals, together with a comparison of the velocity of carbonisation of the coals and of bituminous coal from the Saar basin. During the first half hour of heating there was an evolution of hydrogen sulphide amounting to 8% by volume of the gas produced, which rapidly fell to 2% at the end of 1½ hrs. The ammonia was also evolved very rapidly at first and the yield fell quickly in the same way, accompanied by a diminution in the percentage of carbon dioxide evolved. The residual coke was of the nature of breeze, and was very difficult to quench. It had a calorific value of 4460 to 5947 Cals. per kg. In the tests with mixtures of lignite and bituminous coal, it was found that better results were obtained when the lignite was intimately mixed with the coal by shovelling than when no special care was taken to obtain good admixture. Such careful ad-

mixture is, however, almost impossible in practice. Further, the coke gave less fines when "nut" coal was used than when coal with particles of widely differing sizes was employed.—A. G.

*Coal gas; Composition of — in relation to its utilisation.* R. V. Wheeler. Sheffield Gas Consumers' Assoc. Gas J., 1921, 153, 615–618.

THE character of the flame of a Bunsen burner, which is the type of burner employed in most heating appliances, is entirely dependent on the speed of propagation of flame in the mixture of gas and air that is issuing at the mouth of the burner, and this speed of propagation of flame varies with the composition of the mixture and with the nature of the combustible gas (J., 1920, 94 A, 95 A, 181 A). Speed percentage curves for different gases can be made use of to see how the character of the flame may vary with different gas mixtures. The maximum speed of flame that can be obtained with a given combustible gas and air gives a measure of the tendency of that gas to back-fire. The magnitude of the range of mixtures of a given gas with air lying between the fastest speed mixture and the upper limit mixture gives a measure of the "adjustability" of the gas, and the steepness of the slope of the speed percentage curve on the high-limit side gives a measure of the "adaptability" or ease of adjustment of the gas. There is no reason, on the score of an adaptable mixture of the gas, for trying to place limits on the proportions of combustible gases present in town's gas. The presence of nitrogen, however, has an adverse effect on the ease of adjustment of the gas-air mixture. Large proportions of incombustible gases result in a small margin of adjustment, so that slight variations in the pressure of the gas will cause wide fluctuations in the character of the flame and in the distribution of heat throughout it, with consequent inefficiency of the furnace.—W. P.

*Technical gases; Content of water vapour in —.* G. Sailer. Feuerungstech., 1920, 9, 88–89.

THE author emphasises the importance of estimating the content of water vapour in industrial gases, especially producer-gas. The gas is filtered to remove soot, tar, etc., and then passed through a U-tube filled with calcium chloride or phosphorus pentoxide. Any moisture deposited in the filter tube is estimated by subsequently drying this tube in an air oven and determining the loss in weight. When gases have been washed by a water spray, small globules of water often remain suspended in the gas in an atomised condition, and if a moisture determination is made, it can easily be decided whether free water is present or whether the gas is merely saturated with water vapour.—A. G.

*Water-gas.* L. Vignon. Ann. Chim., 1921, 15, 42–60.

THE mineral matter, and in particular lime, present in coke plays an important part in the composition of the water-gas produced from it. Below 800° C. the formation of calcium carbonate is an important factor in the production of methane. Above 900° C. the carbonate is decomposed, and the calcium oxide formed acts catalytically, favouring the three reactions:  $4CO + 2H_2O = 3CO_2 + CH_4$ ;  $2CO + 2H_2 = CO_2 + CH_4$ ;  $CO_2 + 4H_2 = CH_4 + 2H_2O$ . It is possible by the action of lime to convert carbon monoxide into hydrogen and a mixture of hydrocarbons. Other substances may act as catalysts favouring the interaction of carbon monoxide and water vapour to give methane. With certain of these catalysts (iron, alumina, silica) there is probably an intermediate formation of a carbide, which is decomposed by the water vapour. In other cases hydrogen may be formed, and this then reacts, as above, with the carbon monoxide or dioxide to give methane.

It should thus be possible to prepare a gas approaching coal gas in composition. Water-gas in contact with lime and in the presence of a suitable amount of water vapour at 400° C. gives a gas containing  $H_2$  85–90%,  $CH_4$  5–10%,  $CO$  0–5%. By passing steam over a mixture of coke and lime at 600°–1000° C. a gas containing  $H_2$  80–85%,  $CH_4$  15–20%,  $CO$  0–5% can be obtained. By mixing coal with lime and distilling at 900°–950° C. and then passing sufficient steam over the residual coke at 900°–1000° C. the total gas may have the composition  $H_2$  70–78%,  $CH_4$  15–20%,  $CO$  5–10%. By suitably regulating the reaction the percentage of  $CO$  may be reduced to zero.—W. G.

*Carbon monoxide in air and in flue gases; Determination of small amounts of —.* D. Florentin and H. Vandenberghe. Comptes rend., 1921, 172, 391–393.

Gautier's iodic anhydride method (J., 1898, 490, 603) has the disadvantage that other gases are liable to be present which are also capable of reducing this anhydride. Certain precautions with reference to the apparatus and its manipulation are indicated. The method of Ogier and Kohn-Abrest (Ann. Chim. Analyt., 1908, 169, 218), in which the amount of gas, deprived of oxygen, necessary to cause the appearance of the characteristic absorption bands of carboxyhemoglobin is determined, gave concordant results. The authors recommend each analyst to prepare his own scale. If the gas contains more than 1 pt. of carbon monoxide per 1000, it should be diluted before analysis.—W. G.

*Oil shale; Laboratory testing of — for oil and ammonia yield.* E. L. Lomax and F. G. P. Remfrey. J. Inst. Pet. Tech., 1921, 7, 34–47.

*Oil.*—One kg. is placed in a horizontal iron tube, 20 in. long and 3 in. diam., one end of which is connected with a source of superheated steam, whilst the other end connects with a condenser, a closed receiver, a second condenser, absorption vessels (gas scrubbers), and a graduated aspirator (for measuring gas yield). The iron tube is heated, and as soon as water commences to distil, steam at 180° C. is admitted, and the heating is increased gradually so as to maintain a steady distillation of the oil; when distillation ceases the tube is heated to dull redness for a short time. The distilled oil is examined as to its sp. gr., solidifying point, sulphur content, moisture content, and fractionation. The heavier fractions (above 150° C.) are distilled with the aid of superheated steam. *Ammonia.*—The same apparatus is used, but the shale is placed in the middle portion of the iron tube, the two ends being filled with ignited broken firebrick. The ends of the tube are first heated to dull redness, steam at 180° C. is then admitted, and the whole tube is brought rapidly to a dull red heat; this procedure produces but little oil and a large volume of gas. The vapours are condensed and absorbed in dilute sulphuric acid contained in the receiver and scrubbers, and at the end of the operation the contents of these are mixed and the amount of ammonia determined in the usual way.—W. P. S.

*Gasoline by the charcoal absorption process.* G. A. Burrell, G. G. Oberfell, and C. L. Voress. Chem. and Met. Eng., 1921, 24, 156–160.

A RECENT method for the extraction of gasoline from natural gas is described in which the gas is passed over activated charcoal until absorption is complete, when the gas is replaced by a current of superheated steam to expel the absorbed liquid constituents of the gas. Coconut shell charcoal activated by the steam activation process is the best for the process and should be in granules from 8- to 14-mesh. An advantage of the process is that the

charcoal exhibits "selective" absorption, i.e., when the gas is first passed through, the lighter fractions such as ethane, propane, butane, etc., are absorbed, but as absorption proceeds these constituents are replaced by heavier fractions, and thus the quality of the product can be varied by regulating the extent of the absorption. When the gas first comes in contact with the charcoal the temperature rises owing to the liberation of the latent heat of the condensed vapours, but after a few minutes the rise ceases although absorption is not complete, the heat being utilised to volatilise the lighter fractions condensed previously. In the case of a gas rich in gasoline the temperature rise may be as much as 60° C., and if the gas is passed through a column of the charcoal, a zone at the raised temperature gradually passes along the column in the direction of the gas. The temperature of the inflowing gas has a marked effect on the efficiency of absorption; if the gas is at 10° C. there is 100% absorption, and the absorption decreases constantly as the temperature rises to 300° C., above which there is no absorption at all. The rate of absorption depends upon the richness of the gas; with a gas giving 400 galls. of gasoline per 1,000,000 cub. ft., a gas supply of 40 cub. ft. per hr. per sq. in. of base surface in a 5 ft. column of charcoal is not too high, and gives a back pressure of 1 to 2 lb. After absorption is complete it is necessary to heat the charcoal to at least 200° C. to expel the heavier fractions of the gasoline, and the steam employed for the purpose should be superheated to 250° C. or higher. The vapour is preferably passed through two condensers in series: in the first, just sufficient cooling water is employed to condense the greater part of the steam just below 00° C.; in the second, condensation is completed by the use of an efficient supply of cooling water at about 15° C. The steam distillation does not impair the absorptive property of the charcoal. In comparative tests run on the same days, an oil plant and a charcoal plant extracted respectively 25 and 203 galls. of gasoline per 1,000,000 cub. ft. from the same gas.—L. A. C.

*Sulphur in benzene [petroleum spirit]; Lamp method for determining —. A modified form of apparatus.* J. S. Jackson and A. W. Richardson. J. Inst. Pet. Tech., 1921, 7, 26—34.

In the lamp described, the ordinary wick is replaced by a bundle of capillaries which are "spread" by a central air tube lightly jammed into position; the air supply does not pass through the benzene, so that fractionation is greatly reduced, and the current of air escaping from the top of the air tube produces a slight reduction in pressure, thus assisting the action of the capillaries and enabling them to supply sufficient benzene to maintain a strong non-luminous flame. A mixing chamber is provided above the tops of the capillaries and the luminosity and size of the flame are controlled by the air supply.—W. P. S.

*Petroleum spirit; Analysis of —. Critical temperature of solution of hydrocarbons in aniline.* N. Chercheffsky. Ann. Chim. Analyt., 1921, 3, 53—55.

The method described by Chavanne and Simon (J., 1919, 619 A, 672 A) has been published previously by the author (J., 1910, 681).—W. P. S.

*Combustion of carbon.* Berger and Delmas. See VII.

*Soaps from paraffin wax etc.* Schrauth and Friesenhahn. See XII.

#### PATENTS.

*Suction gas generators.* W. E. Scrivner, Assee. of S. Phillips. E.P. 132,269, 6.9.19. Conv., 6.9.18.

The fuel feed hopper and the generator body are made of stout galvanised iron, the generator being lined with asbestos, with an inner course of fire-brick in the furnace zone. A galvanised iron wire is coiled spirally round the generator body and welded to it; a stream of water is fed on to the wire spiral to generate steam. Fuel is supplied through a central depending shoot, concentric with the furnace, and the gases are taken off from a side pipe in the cowl, of which the hopper forms a part.—A. G.

*Blue water-gas; Plant for the manufacture of — in conjunction with coal gas.* J. Lowe. E.P. 149,923, 2.12.19. Conv., 19.8.19.

Blue water-gas is made in twin generators, so arranged that whilst one is being blown with steam the other is being blown with air. The blue water-gas is either passed into coal being carbonised in the retorts of a separate installation or is mixed with coal gas in the gas main.—A. G.

*Combustion gases of a fuel; Apparatus for utilising the — for generating steam mixed with these gases and, if desired, a percentage of pure steam.* F. H. E. Marden. E.P. 158,326, 29.10.19.

Liquid fuel is sprayed at high pressure into a vaporiser, where it is mixed with compressed air supplied through a non-return valve. The mixture is ignited by an electric spark and the gases of combustion pass through headers and side-tubes surrounded by water, which thus becomes heated. The vertical side-tubes are open at the end and project above the water level; the gases of combustion can thus be mixed with the steam generated. By means of a by-pass, the hot gases can be passed into an ordinary fire-tube boiler, thus generating steam unmixed with the products of combustion.—A. G.

*Producer gas plants; Generators for —.* T. H. White. E.P. 153,409, 14.11.19.

The producer comprises a combustion chamber of rectangular cross-section, the coke being supported by fire-bars and a superimposed cylindrical structure. The ratio of the area of the grate or complete combustion chamber to that of the superposed converter chamber is 2.8 to 1. It is claimed that clinkering is largely reduced with such a plant.—A. G.

*Combustible gaseous compositions especially for use in cutting, welding, and the like.* R. H. Brownlee and R. H. Uhlinger. E.P. 153,633, 5.11.19.

A suitable gaseous fuel for the purpose specified consists of over 75% of hydrogen, over 5% of hydrocarbon, and not more than 10% of carbon monoxide. For special purposes, and where it is desired to avoid oxidation of the metal, the gas may consist of about 87% of hydrogen, about 9% of hydrocarbon, and 4% of carbon monoxide.—A. G.

*Gas [from garbage]; Process for making —.* J. U. McDonald. U.S.P. 1,367,321, 1.2.21. Appl., 10.3.20.

Wet garbage is placed in a vertical retort and heated at an intermediate zone so that it is carbonised and rendered incandescent at that point. The moisture and gas distilled from the upper layers pass down through the incandescent zone, and all the combustible constituents are thereby converted into fixed gas.—W. F. F.



*Gas producer for obtaining low temperature tar.* Deutscher Industrie-Ofenbau, G.m.b.H. G.P. 327,095, 31.8.17.

THE distillation gases are withdrawn through a central gas outlet pipe fixed to the revolving grate. They enter the upper end of the outlet pipe, within the producer shaft, through a perforated hood provided with sloping metal plates between the perforations.—C. A. C.

*Gases; Collection of — in chambers.* C. C. Carpenter. E.P. 158,758, 3.12.19.

To ensure uniformity of composition of the contents of a gas-holder, a portion of the gas is withdrawn to a compressor and is re-introduced through a nozzle, forming an injector, thus producing thorough agitation of the contents of the holder.

—A. G.

*Hydrocarbons; Process for converting higher molecular — into lower molecular ones.* Naam. Vennoten. Nederlandsche Lichte Olie Maatschappij, Assoes. of N. V. Hollandsche Benzol en Benzine Maatschappij. E.P. 135,197, 12.11.19. Conv., 12.11.18.

A MIXTURE of high-molecular hydrocarbon (tar oil or petroleum) vapour and superheated steam is passed up a vertical retort packed with coke or the like to the extent of  $\frac{1}{2}$  to  $\frac{3}{4}$  of its height. The retort is heated externally so that the middle zone of the packing material is maintained at 500°–1200° C., while the top and bottom zones are heated to a temperature 300° C. lower than that of the middle zone, the height of which is from  $\frac{1}{4}$  to  $\frac{1}{2}$  of the total height of the retort.—L. A. C.

*Petroleum and analogous oils; Process of distilling and cracking —.* A. J. Paris, jun. U.S.P. 1,367,828, 8.2.21. Appl., 27.12.16.

A MIXTURE of a hydrocarbon gas and the oil under treatment is introduced into a body of molten metal.

—L. A. C.

*Hydrocarbon distillates; Process for the treatment of —.* W. J. Hoffman, Assr. to U.S. Motor Fuel Corp. U.S.P. 1,367,968, 8.2.21. Appl., 9.8.17.

A SOLUTION containing picric acid and wood alcohol impregnated with acetylene gas is added to the hydrocarbon.—L. A. C.

*Illuminating gas; Apparatus for generating —.* A. A. Birkholz. U.S.P. 1,355,530, 12.10.20. Appl., 10.11.16. Renewed 3.8.20.

SEE G.P. 299,546 of 1915; J., 1918, 456 A.

*Hydrocarbons; Process for conversion of — into hydrocarbons of lower boiling point.* R. Fleming. E.P. 135,854, 26.11.19. Conv., 17.11.16.

SEE U.S.P. 1,324,766 of 1919; J., 1920, 99 A.

*Briquette presses.* C. W. G. Clewlow. E.P. 158,728, 18.11.19.

*Gas producers; Supply of blast to [and grates of] —.* J. F. W. Pasley. E.P. 158,786, 30.12.19.

*Combustion furnaces.* U.S.P. 1,354,741. See I.

*Reaction column.* G.P. 327,164. See I.

*Asphaltic cement.* E.P. 158,674. See IX.

*Fatty acids from hydrocarbons.* G.P. 307,048. See XII.

## IIa.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Wood distillation. III. Distillation of some Mysore and Baroda woods and of certain waste products.* J. J. Sudborough, H. E. Watson, M. N. Bahuguna, K. R. Dotiwala, M. G. Kekre, M. O. Krishnaswamy, M. K. Narasimhan, and S. M. Nilikani. J. Indian Inst. Sci., 1920, 3, 281–292.

THE distillation of a further 23 species of Mysore woods and 8 species of Baroda woods gave results similar to those previously obtained (cf. J., 1920, 327 A), i.e., the yields of acetic acid and methyl alcohol were generally lower than those obtained on distilling European and American hardwoods. The yields from the Baroda woods were, as a rule, lower than those from the corresponding Mysore woods. Undried coconut shells yielded 4.90–6.90% of acetic acid and 0.66–1.54% of methyl alcohol; wattle wood (*Acacia decurrens*), the bark of which is utilised for the extraction of tannin, gave yields which compared favourably with those obtained from hardwoods; myrobalan kernels, the husks from the dry pods of the gold mohur tree (*Poinciana regia*), and bamboo gave yields comparable with those from the South Indian jungle woods.—L. A. C.

*Tar from coconut shells.* Sudborough and others. See III.

### PATENTS.

*Vertical retorts for the destructive distillation of coal, shale and the like.* S. Jones and J. D. Macdonald. E.P. 109,800, 23.8.17. Conv., 15.7.16.

NEAR the bottom of the retort and external to it is a special valve consisting of two smooth plates with a drum in between. The drum has two pockets, one on each side of the centre, and each plate has an opening opposite to the position of one pocket, such opening in one plate being placed opposite the neck of the feed hopper, whilst the opening in the other plate affords communication between the second pocket in the drum and a short conduit leading into the bottom of the retort. By rotating the valve drum through 180° the material supplied to one pocket from the hopper is caused to pass through the valve into a conduit from which it is forced to the top of the retort by means of an archimedean screw. The hood of the retort forms the gas and coke exit and is water-sealed, the coke being removed along a channel by push-plate conveyors.

—A. G.

*Carbonaceous material; Apparatus for distilling —.* W. P. Perry. E.P. 153,394, 10.11.19.

AN apparatus for continuously distilling and gasifying carbonaceous material comprises a distilling chamber having feed appliances at the top and apparatus for ash removal at the bottom. The chamber has a number of openings in the walls, or louvres on two opposite sides, a chamber on one side opening into the distilling chamber at the lower part for distributing hot gases into the mass through the openings, and a chamber on the other side for receiving the gases and vapours through the openings from the distilling chamber. Outlets are provided from the collecting chamber for conducting away the gases, vapours, and liquid distillates.—A. G.

*Carbonisation of coal, lignite, shale, and like materials; Apparatus for —.* G. F. Bale. E.P. 158,622, 4.11.19.

THE retort comprises an inner and an outer vertical shaft, separated by a heating space. The central shaft is surmounted by an annular cast-iron trunk which serves to collect distillate and to prevent it from returning by gravity to the inner

shaft. A sliding box is operated by a ram under the central chamber, so that a predetermined quantity of carbonised material can be removed from the retort at any time.—A. G.

*Carbon; Process of activating* — J. C. Woodruff. U.S.P. 1,368,937, 15.2.21. Appl., 8.5.19.

CARBON is activated by oxidation by continuously agitating it whilst conveying it through a reaction zone, wherein it is subjected to the combined action of steam and hot combustion gases.—A. B. S.

*Incandescence gas mantles; Manufacture of* — W. Marshall. E.P. 153,779, 19.12.19.

CELLULOSE acetate employed in the manufacture of incandescence gas mantles is subjected to a process of partial or complete de-acetylation by treating the material or the mantle with solutions of the hydroxides, carbonates, or silicates of sodium or potassium, or a mixture thereof, at a temperature below 212° F. (100° C.) for 10–30 mins. The mantle or material is then impregnated with thorium and cerium nitrates in the usual manner.—J. S. G. T.

*Electric arc lamps or projectors and carbons or electrodes therefor*. C. B. Burdon. From Siemens-Schuckertwerke, G.m.b.H. E.P. 153,330, 6.11.19.

AN anode for use in a high-effect projector is composed of a carbon tube having a wick or core containing cerium or a non-pyrophoric alloy thereof, mixed in a finely divided state with pulverised carbon or graphite, and a binding medium such as tar. The external diameter of the anode is smaller than the normal crater diameter of a pure carbon anode for the same load.—J. S. G. T.

### III.—TAR AND TAR PRODUCTS.

*Tar from coconut shells. Wood distillation. IV.* J. J. Sudborough, H. E. Watson, and M. K. Narasimhan. J. Indian Inst. Sci., 1920, 3, 293–306.

THE tar from coconut shells yielded on distillation about 24% of aqueous distillate with 8% of acetic acid and 44% of oils volatile below 260° C. which gave 30% of phenolic substances soluble in sodium hydroxide solution. These substances on fractionation yielded 6.2% of phenol and 5.86% of creosote, b.p. 200°–230° C., containing 0.43% of guaiacol, the yields all being calculated on the weight of the tar. The sp. gr. of the creosote, however, was lower than that required by either the British or U.S. Pharmacopœia.—L. A. C.

*Tar-oils; Determination of acids in* — A. Lazar. Chem.-Zeit., 1921, 45, 197–199.

FOR the estimation of creosote (tar acids), especially in low-temperature tar oils, a gravimetric method is recommended. The oil is treated with 5% sodium hydroxide, the alkaline creosote solution decomposed with dilute sulphuric acid, and the creosote extracted with ether. The ethereal solution is first distilled, after which the last traces of ether are removed by careful heating over a small flame. In the differential method, in which the oil is treated with sodium hydroxide, and the increase in volume of the latter is measured, addition of benzene to prevent solution of substances other than creosote may result in absorption of part of the benzene itself. The benzene should first be added to the sodium hydroxide, and the oil then introduced. After reading the volume the alkaline creosote solution is distilled, and a correction is made for the benzene which is recovered from it.—W. J. W.

*Phenol; Characteristic reaction of* — G. Rodillon. J. Pharm. Chim., 1921, 23, 136–137.

TEN c.c. of a solution containing phenol is treated with a drop of 10% sodium nitrite solution and the mixture is poured on the surface of concentrated sulphuric acid. A coloured zone develops at the point of contact of the two liquids, the upper part of the zone being red and the lower green.—W. P. S.

*Phenol and some mineral solutions; Miscibility of* — Analytical applications. R. Dubrisay. Bull. Soc. Chim., 1921, 29, 78–88.

A DETAILED account of work already published (cf. J., 1919, 37 A, 103 A; 1920, 542 A, 766 A).—W. G.

*Sulphonation of anthraquinone; Catalytic action of mercury in the* — A. Roux and J. Martinet. Comptes rend., 1921, 172, 385–387.

IN the absence of mercury the sulphonation of anthraquinone yields the  $\beta$ -sulphonic acid, but in the presence of mercury at a lower temperature the product is the  $\alpha$ -acid. It is shown that in both cases the first product is the  $\alpha$ -acid, but that at the higher temperature required in the absence of mercury the velocity of transformation of the  $\alpha$ -acid into the  $\beta$ -acid is as great as the velocity of sulphonation. In the presence of mercury, but at the higher temperature, the product is also the  $\beta$ -acid, and a higher yield is obtained.—W. G.

*Phenanthrene; Bromine compounds of* — H. Henstock. Chem. Soc. Trans., 1921, 119, 55–61.

THE bromination of phenanthrene in carbon tetrachloride solution using four atoms of bromine for 1 mol. of phenanthrene gave 31% of 8-bromophenanthrene and 30% of 2(?)-bromophenanthrene dibromide, which decomposed at 100° C. to hydrogen bromide and 2(?) 10-dibromophenanthrene. The position of the 10-bromine atom was demonstrated by the oxidation of the compound to a dibromophenanthrone, instead of to a quinone which would have been formed were the 10-position unoccupied. The position of the second bromine atom is uncertain, the only evidence being that 2-ethoxyphenanthrene similarly treated gave only a 10-monobromo-substitution product.—G. F. M.

*Carbazole; Oxidation of* — W. H. Perkin, jun., and S. H. Tucker. Chem. Soc. Trans., 1921, 119, 216–225.

BY the action of potassium permanganate on carbazole in boiling acetone solution three substances are produced, viz., two crystalline compounds, melting at 220°–221° C. and 265° C. respectively, and an amorphous substance. The crystalline compounds are isomeric dicarbazyls having the molecular formula  $C_{24}H_{16}N_4$ . All three substances can be nitrated and brominated and react under certain conditions with acetic anhydride. They differ from carbazole in that they do not form picrates.—G. F. M.

*Tetrahydronaphthalene derivatives from  $\gamma$ -phenyl-fatty acids*. Kon and Stevenson. See XX.

#### PATENTS.

*Coal tar; Process of treating* — W. Anderson and J. Meikle. E.P. 153,337, 31.10.19.

THE tar is preheated and injected by means of superheated steam in excess of that required to vaporise the tar into a length of tubing, e.g., 72 ft. of 5 ins. diam., at such a rate that 20 tons of tar is distilled in 24 hrs. The products pass into a chamber wherein carbon is deposited, and thence into a series of condensers for fractional condensation of the products of the reaction, which consist of gas, carbon, a liquid similar in appearance to coal

tar creosote, and a reddish-brown solid of resinous appearance, of which about 40% is soluble in alkalis and about 9% in acids.—L. A. C.

*Still [for distillation of pitch].* H. P. Hird. U.S.P. 1,368,149, 8.2.21. Appl., 27.6.19.

THE still has a circular cross-section and a curved longitudinal axis, the ends of the still being turned upwards so as to be above the normal level of the pitch. Openings with removable lids are provided at each end for removing the residue from the still, and an inlet is attached to one end and a vapour outlet to the other.—L. A. C.

*Naphthalene and its derivatives; Manufacture of condensation products of — with aralkyl halides.* Farbenfabr. vorm. F. Bayer und Co. G.P. 301,713, 29.1.16.

THE condensation is carried out in presence of small amounts of iron or its compounds. By the condensation of methyl-naphthalene with benzyl chloride an odourless oil is formed, soluble in benzene and petroleum spirit, whilst naphthalene and xylol chloride yield an odourless, yellowish-red oil, b.p. 220°–380° C. at 25 mm. The products may be used for technical and medicinal purposes, e.g., for skin diseases as substitutes for tar.—C. A. C.

*Converting hydrocarbons.* E.P. 135,197. See IIA.

#### IV.—COLOURING MATTERS AND DYES.

*Indigoid dyestuffs; Colour of —.* J. Martinet. Rev. Gén. Mat. Col., 1921, 25, 17–18.

FACTS such as the increase in the depth of colour in passing from brown-red isoindigo to violet-red indirubin and blue indigo, the existence of a similar relationship in the case of oxindigo, thioindigo, and indigo, and the diminution in the depth of colour produced by substituents, such as methyl, hydroxyl, and amino groups and the halogens in the 6.6' positions, could be predicted by the application of the following rules. The union of an auxochrome and a chromophor by means of a chain of conjugated double bonds between carbon atoms leads to an increase in the depth of colour (bathochromic effect). The direct union of a chromophor and an auxochrome decreases the depth of colour (hypsochromic effect). In the case of isoindigo the auxochrome NH-groups are directly attached to the chromophor CO groups and are consequently in an unfavourable position for the production of colour, whereas in indigo the NH group exerts its maximum effect because it is attached to the CO group by a benzene nucleus on one side and by a carbon atom, which forms part of a chain with double conjugated bonds, on the other. The formula of indirubin is intermediate between the two, and this fact is reflected in its colour. The increase in the depth of colour in passing from oxindigo to thioindigo and indigo is a bathochromic effect due to the increasing power of the auxochromes C<sub>6</sub>H<sub>5</sub>O, C<sub>6</sub>H<sub>5</sub>S, C<sub>6</sub>H<sub>5</sub>NH<sub>2</sub>, and is in agreement with experience in other series. The effect of two auxochromes is cumulative when in the *p*-position, but they act in contrary directions when in the *m*-position with respect to one another. This also is in agreement with experience in other series, but is particularly well exemplified in the indigoid series, for all 5.5'-derivatives are deeper in colour than the corresponding 6.6'-derivatives. (Cf. J.C.S., April.)—F. M. R.

*o-Chlorodinitrotoluenes.* II. G. T. Morgan and L. A. Jones. Chem. Soc. Trans., 1921, 119, 187–192.

2-CHLORO-4.6-DINITROTOLUENE, recently isolated from the nitration product of 2-chloro-4-nitro-

toluene (J., 1920, 623 A), has now been obtained to a similar extent in the nitration of 2-chloro-6-nitrotoluene. 6-Chloro-2.4-tolylenediamine, which it yields on reduction with alcoholic stannous chloride, yields azo- and bisazo-derivatives of the chrysoidine series resembling those obtained from 6-chloro-3.5-tolylenediamine (Chem. Soc. Trans., 1902, 81, 97). The action of ammonia and primary amines, such as methylamine or aniline, on 2-chloro-4.5-dinitrotoluene leads to the displacement of the 5-nitro-group by the aminic radicle, whilst in the case of 2-chloro-5.6-dinitrotoluene it is the 6-nitro group which is displaced.—G. F. M.

*Catechin; Constitution of —.* III. Synthesis of acacatechin. M. Nierenstein. Chem. Soc. Trans., 1921, 119, 164–170.

IN a previous communication (cf. J., 1920, 685 A, 777 A) it was provisionally suggested that catechin is 2.4.6.3'.4'-pentahydroxy-3-phenylchroman. This substance has now been synthesised from 3.4.2'.4'.6'-pentamethoxydiphenylmethyl chloromethyl ketone, which was converted by the action of aluminium chloride into 4.6.3'.4'-tetramethoxy-3-phenylchroman-2-one. This was reduced to the corresponding hydroxy-compound, which proved to be identical with acacatechin tetramethyl ether, and on demethylation it gave the above-mentioned pentahydroxyphenylchroman, identical in all respects with acacatechin.—G. F. M.

#### PATENTS.

*Dye substitute.* A. L. Clapp, Assr. to The Metalite Co. U.S.P. 1,367,862, 8.2.21. Appl., 20.12.15.

THE product obtained by dissolving leather in a hot aqueous solution of caustic soda (proportions: 100 lb. of leather, 15 lb. of caustic soda, and 1000 qts. of water) may be used for dyeing fabrics, pulp products, paper, leather, and similar substances.—C. A. C.

*Dye.* S. M. Tootal. U.S.P. 1,367,930, 8.2.21. Appl., 22.4.20.

A PREPARATION for dyeing silk or wool in mixed fabrics whilst leaving the cotton unaffected is composed of a weakly acid dyestuff, a gelatinous substance, and a weak organic acid.—C. A. C.

*Indigo and other vat dyestuffs; Process for obtaining — in a finely divided form.* C. H. Boehringer Sohn. G.P. 326,573, 9.10.17.

THE separation of the dyestuff from solutions of indigo white or analogous compounds of other vat dyes, is carried out in presence of salts of bile acids. On blowing air through a solution of sodium-indoxyl containing 5–10% of sodium cholate, indigo separates in a very fine form, particularly suitable for vat working.—J. H. L.

*Lake colours.* U.S.P. 1,358,007. See XIII.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Wool fabrics; Formation of stains on, and tendering of — due to faulty carbonisation.* P. Heermann. Textilber., 1921, 2, 106–107.

LARGE and small dark stains often found in coloured woollen dress materials which have been carbonised are due to the use of too strong an acid or too high a temperature during carbonisation. Where the material is stained, the wool is tender and under the microscope is shown to be completely destroyed.—A. J. H.

*Cellulose; Methylation of* —. III. Homogeneity of product and limit of methylation. W. S. Denham. Chem. Soc. Trans., 1921, 119, 77–81. (Cf. J., 1913, 974; 1914, 1034.)

HIGHLY methylated celluloses were obtained by treating cotton with sodium hydroxide solution and then with methyl sulphate, and repetitions of this process, variously modified, have yielded a product containing 44.6% of methoxyl, a value approximating to that theoretically required for trimethyl-cellulose. This is regarded as the limit of methylation and as far as could be ascertained from its behaviour to Schweitzer's reagent, in which it was quite insoluble whilst lower methylated celluloses were partially soluble, it was apparently homogeneous.—G. F. M.

*Nitrocellulose solutions.* Herzog and others. See XIII.

*Pyroxylin compositions.* Lorenz. See XV.

*Cellobiose.* Haworth and Hirst. See XVII.

#### PATENTS.

*Balloon or like fabrics.* C. A. Cleghorn. E.P. 158,366, 4.11.19.

FABRIC prepared with bird-lime according to E.P. 142,160 (J., 1920, 444 A) does not adhere well to a non-absorbent fabric. To avoid this difficulty, fabric is coated with a thin layer of Turkish bird-lime (E.P. 130,379; J., 1919, 730 A), heated to 180° F. (82° C.) to expel moisture, coated with a rubber solution, and placed in contact with a second sheet of fabric. For preparing single fabrics the rubber is dried and vulcanised, or coated with shellac varnish, or replaced by shellac varnish; 0.1% of mercuric chloride incorporated with the bird-lime is a suitable preservative.

—A. J. H.

*Retting flax or the like; Process of* —. B. S. Summers. E.P. 158,807, 1.5.20.

FLAX is retted in open tanks by the usual process, except that the retting liquor is continuously withdrawn, filtered, diluted so that its oxygen absorption value, as measured by means of a standard solution of potassium permanganate, is maintained constant, and returned to the retting tanks. By this means the increase of harmful putrefying bacteria is checked.—A. J. H.

*Fabrics and packings, such as bags; Process of protecting — against chemical corrosive action.* A. Bruno. U.S.P. 1,367,177, 1.2.21. Appl., 21.11.18.

FINELY-GROUND chalk and a colloidal agglutinant diluted with water are applied to fibres to be used for fabrics, e.g., bags, to contain corrosive materials.—A. de W.

*Fibres of the agave and similar fleshy plants; Method of liberating the* —. R. Quijano. U.S.P. 1,368,750, 15.2.21. Appl., 4.8.19.

THE fibres are separated by crushing the cut plants, soaking the leaves in a bath capable of removing the easily soluble gums, washing, and boiling with a reagent capable of removing the remaining gums.

—A. J. H.

*Fibrous plants, such as the stinging nettle and the like; Pre-treatment of woody — for the dry breaking process.* Nessel-Anbau-Ges.m.b.H., and A. Hoermann. G.P. 326,489, 17.9.18.

BEFORE being dried the plants are impregnated with substances which inhibit the action or development of bacteria, e.g., antiseptics, per-salts, and diastatic enzymes, especially diastatic malt preparations.—J. H. L.

*Textile fibres; Preparation and improvement of* —. E. Herzinger. G.P. 328,595, 21.1.19.

THE material is treated with alkali sulphide solution, squeezed, and without being rinsed is introduced into an acid bath.—J. H. L.

*Fibrous plants; Process for the simultaneous production of textile fibres and paper pulp from* —. Nessel-Anbau-Ges.m.b.H. G.P. 328,596, 2.2.18.

THE plant material is treated with suitable reagents under pressure or in concentrated form, in presence of dissolved or emulsified vegetable, animal, or mineral oils or fats, or halogen derivatives of hydrocarbons, which serve to protect the bast fibres. After the treatment the material is rinsed with water or suitable solutions, and neutralised if necessary with acids or acid salts; the bast fibres are separated from the cellulose fibres derived from the woody tissue, and the latter can be used for the manufacture of paper.—J. H. L.

*Straw and similar materials; Production of textile fibres from* —. G. Strauss. G.P. 328,597, 5.2.20. Addn. to 323,669 (J., 1920, 744 A).

HYDROGEN peroxide is added to the steeping solution.—J. H. L.

*Wool, woollen yarn, and woollen fabrics; Process for the removal of fat from raw* —. S. Aschkenasi. G.P. 329,008, 1.6.16.

THE soap solutions commonly employed for the purpose are neutralised or acidified with lipolytic substances, viz., hydricinoleic acid, naphthalene-sulphonic acid, Twitchell's reagent, contact hydrolysis, or sulphonated fats. The injurious action of alkali on the wool is thus avoided, and the fatty acids remain dissolved.—J. H. L.

*Material for bandages and filter cloths; Manufacture of adsorptive and absorptive* —. R. Marcus. G.P. 329,310, 8.10.15.

TEXTILE material is soaked in a colloidal solution of silicic acid and then dried. Other soluble substances, as silver, iron, and other salts, may be applied together with the silicic acid.—C. A. C.

*Fabrics; Composition for removing iron rust from* —. J. E. Gliott. U.S.P. 1,363,714, 15.2.21. Appl., 8.2.19.

IRON rust is removed from fabrics by treatment with a solution of hydrofluoric acid mixed with an amount of borax in excess of the chemical equivalent.—A. J. H.

*Acetylcellulose; Process for preparing layers of — for coating materials.* A. Eichengrün, Assr. to American Cellone Co. U.S.P. 1,357,447, 2.11.20. Appl., 11.4.10. Renewed 2.4.20.

A SOLUTION is prepared by treating cellulose acetate in the cold with two solvents of which, when used alone, one is capable of dissolving cellulose acetate in the cold but the other only on heating.

*Viscose; Process for producing threads from* —. C. A. Hutterling and E. Rittenhouse, Assrs. to The Acme Woolen-Cotton Mills Co. U.S.P. 1,367,603, 8.2.21. Appl., 3.12.19.

VISCOSE is forced in multiple streams into a precipitating bath containing an aqueous solution of sodium bisulphate and molasses, and the resulting filaments are spun, whereby a soft and open thread is obtained.—A. J. H.

*Cellulose and artificial resin, lacquer, etc.; Treatment of wood or cellulosic materials for the production of —.* R. Hartmuth. G.P. 328,783, 27.11.19. Addn. to 326,705 (J., 1921, 144 A).

INORGANIC or organic acids, or acid substances, with the exception of hydrochloric acid, may be employed as catalysts. The following catalysts for the phenolic digestion process are mentioned:—ammonium salts, stannous, stannic, aluminium, and zinc chlorides, silicon tetrachloride, and nitro- and chloro-phenols.—J. H. L.

*Cellulose; Production of —.* Köln-Rottweil-A.-G. G.P. 329,566, 16.1.20.

WOOD and other cellulosic substances are treated at high temperatures with glycol or mineral acid esters of polyhydric alcohols, e.g., dichlorohydrin, with or without the aid of high pressures, or catalysts, such as sulphuric or hydrochloric acid or zinc chloride. The esters also accelerate the action of polyhydric alcohols when used with the latter, and they facilitate the solution of encrusting substances in acetone, acetic acid, etc.—J. H. L.

*Wood-like article of manufacture and process of making the same.* T. B. Mohler and O. Ludekens, Assrs. to The Moludite Co. U.S.P. 1,357,647, 2.11.20. Appl., 22.9.19.

CRUSHED barley is boiled with water, then fermented with yeast, the product treated with a small quantity of precipitated iron carbonate and mixed with paper or with the pulp obtained by digesting straw or the like with alkali, so as to obtain a plastic dough, which is moulded and baked.

*Paper yarn and fabrics and other cellulose threads; Process for obtaining — of wool-like character.* E. Herzinger. G.P. 326,806, 21.1.19.

THE material is treated with solutions of alkali sulphides, whereby it is rendered soft and wool-like to the touch and partially bleached. On pressing it becomes compact like a true textile fabric, and remains soft.—J. H. L.

*Waste paper; Apparatus for (a) decolorising, (b) decolorising and disintegrating —.* W. Abele. G.P. (a) 328,671, 17.8.19, and (b) 328,731, 8.1.20. Addns. to 305,343 (J., 1918, 410 A).

(A) THE heater is situated within the decolorising vessel. (B) The liquid is circulated through the material by means of a steam injector pump mounted outside the vessel.—J. H. L.

*Paper and other cellulose products; Production of absorbent —.* Zentralstelle für wiss.-techn. Untersuchungen G.m.b.H. G.P. 328,788, 27.11.19.

THE pulp, before or after it has been given its final shape, is dried in a frozen state. The fibres are thus prevented from adhering together, and the products possess great absorbent capacity.

—J. H. L.

*Cork; Production of shaped pieces of — of low specific gravity, by heating cork fragments in moulds.* Grünzweig und Hartmann, G.m.b.H. G.P. 326,882, 7.5.13.

THE material is heated between 250° and 300° C. by placing the mould in a heating space which, throughout the whole heating process or during the latter part, has a constant maximum temperature of 360° C. Moulded forms having density as low as 0.05 may be produced.—J. H. L.

*Wool substitute from cellulose solutions; Process for manufacturing a —.* Glanzfäden A.-G. E.P. 135,206, 13.11.19. Conv., 14.7.17.

SEE G.P. 312,304 of 1919; J., 1920, 13 A.

*Pyroxylin compositions.* E. I. du Pont de Nemours and Co., Asses. of E. M. Flaherty. E.P. 133,972, 17.10.19. Conv., 16.3.18.

SEE U.S.P. 1,321,611 of 1919; J., 1920, 14 A.

*Pyroxylin solvent and pyroxylin compositions containing the same.* E. M. Flaherty. E.P. 158,586, 8.7.19.

SEE U.S.P. 1,323,624 of 1919; J., 1920, 60 A.

*Fibrous material; Process for the manufacturing of — from wood or the like.* F. K. L. Schouten, Assr. to J. C. Van Wessem. U.S.P. 1,367,896, 8.2.21. Appl., 1.6.18.

SEE E.P. 117,086 and 122,812 of 1918; J., 1919, 496 A, 625 A.

*Pulp for paper-making purposes and the like; Apparatus for washing —.* S. Milne. U.S.P. 1,367,707, 8.2.21. Appl., 24.7.17.

SEE E.P. 109,594 of 1917; J., 1917, 1174.

*[Pulp] digesters; Method of short-time digestion in sulphite or sulphate —.* J. K. Ruths, Assr. to Aktiebolaget Vaporackumulator. U.S.P. 1,368,577, 15.2.21. Appl., 10.3.19.

SEE E.P. 144,084 of 1919; J., 1920, 541 A.

*Drying sheet material [paper etc.]; Process of —.* O. Minton. E.P. 142,809, 3.5.20. Conv., 6.10.14.

SEE U.S.P. 1,147,808 of 1915; J., 1915, 901.

*Osmotic diaphragm.* U.S.P. 1,368,227. See I.

*Membrane filters.* G.P. 329,060 and 329,117. See I.

*Sulphite-cellulose waste liquors.* G.P. 329,111. See XVIII.

*Destroying cellular structures.* U.S.P. 1,355,476. See XIX a.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Aniline Black dyeing.* J. Schmidt. Textilber., 1921, 2, 105—106.

THE loss of strength of fabrics dyed with Aniline Black under the most favourable conditions is 5—15%. When "aniline salt" is used in the padding liquor, the free mineral acid which it contains is neutralised with aniline oil, and acetic, formic, or lactic acid is added; 30 pts. of sodium chlorate is required to oxidise 100 pts. of aniline salt. Attempts to replace sodium chlorate by perborates or peroxides were unsuccessful because the resulting mixtures were unstable. Copper salts used as catalysts help to tender the fabric. The strength of the fabric is unaffected by the addition of 1—2% of acetic, formic, or lactic acid to the padding liquor, but tartaric acid produces tendering. The presence of alkaline-earth salts of acetic and formic acids in the padding liquor preserves the fabric, since a metallic hydroxide is formed within the fibres. Magnesium chloride (30 c.c. of a 6% solution per 1000 c.c. of padding liquor) is a suitable hygroscopic substance. The ageing-chamber should be well ventilated so as to remove acid vapours; the temperature should not exceed 30°—35° R. (38°—44° C.) and the atmosphere contained therein should be 50% saturated with moisture. After chroming, the fabric should be soured, washed twice, and soaped.—A. J. H.

*Sodium sulphide in sulphide dye-baths; Determination of —.* W. Herbig. *Z. angew. Chem.*, 1921, 34, 89–92.

THE results obtained in the estimation of pure sodium sulphide with zinc sulphate and sodium nitroprusside agree well with those obtained by titration with iodine. The former method also gives approximately concordant results with those of control experiments in the case of the estimation of the residual sodium sulphide in used dye-baths, but with freshly prepared sulphide dye solutions the zinc sulphate method gives very irregular results. In the preparation of the sulphide dye solution, owing to the interaction of the dye with sodium sulphide, as much as 50–80% of the latter is consumed in certain cases. It would appear that the addition of soda in the preparation of sulphide dye solutions has only a slight influence on this consumption of sodium sulphide; nevertheless, it is recommended that sulphide dyes should be worked into a paste with soda, prior to the addition of sodium sulphide.—F. M. R.

#### PATENTS.

*Fibres, threads or fabrics [of cellulose acetate]; Dyeing or colouring —.* British Cellulose and Chemical Manuf. Co., J. F. Briggs, and C. W. Palmer. E.P. 158,340, 31.10.19.

AFTER treatment with a solution of ammonium thiocyanate, cellulose acetate has an increased affinity for all classes of dyestuffs. Fabrics containing cellulose acetate are immersed for 2–60 mins. at ordinary temperature in a 5–25% solution of ammonium thiocyanate, thoroughly washed, and dyed in the usual manner. In some cases, the ammonium thiocyanate may be added to the dye-bath. Sodium, potassium, and calcium thiocyanates may also be used.—A. J. H.

*Dyeing; Process of —.* H. B. Smith, Assr. to Surpass Chemical Co. U.S.P. 1,368,298, 15.2.21. Appl., 30.1.20.

COTTON material is treated in a dye bath to which is subsequently added an alkaline oxidising agent so as to remove the motes, shives, and/or leaf which the material contains.—A. J. H.

*Patterns resembling marble or battick effects; Production of —.* C. Jäger, G.m.b.H., and R. W. Carl. G.P. 329,173, 14.5.19.

A SOLUTION of a dye soluble in oil or fat is suitably disposed on the surface of water or other liquid and the material to be coloured is laid thereon and then removed and dried.—J. H. L.

*Overprinting fabrics and yarns [; Machines for —].* Calico Printers Assoc., Ltd., F. and R. Ashton, and G. Mellor. E.P. 158,407, 14.11.19.

*Detergent and fulling agent.* G.P. 328,812. See XII.

### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Lead sulphuric acid chamber; Methods of introducing nitrogen compounds into the —.* G. Gianoli. *Giorn. Chim. Ind. Appl.*, 1921, 3, 11–12.

THE author discusses the advantages and disadvantages attaching to each of the different methods which have been suggested for supplying the nitrous compounds required in the manufacture of sulphuric acid, with special reference to the investigations of Lemaitre (J., 1920, 655 A) and to his own proposal to employ in the Glover tower a mixture of

sulphuric and nitric acids obtained by pouring sulphuric acid of 20° B. (sp. gr. 1.16) into concentrated sodium nitrate solution and removing the sodium bisulphate, which crystallises out on cooling (Sixth International Congress of Applied Chemistry, 1906).—T. H. P.

*Nitrogen; Fixation of atmospheric — by the silent electric discharge process.* I. C. F. Harding and K. B. McEachron. *Amer. Inst. Elect. Eng.*, 9.4.20. [Advance copy.]

THE results of experiments on the production of nitric acid by the passage of a silent electric discharge through fixed volumes of air or other nitrogen-oxygen mixtures were recorded by Spiel in a paper not generally accessible (Thesis, Vienna Technical High School, 1909). Spiel found that during the discharge the pressure gradually fell to a minimum, and subsequently a reversal of the reaction took place, the pressure gradually returning to the initial or even a greater value. At the reversal point the concentration of nitric oxide was about 5 to 6% by vol., but at the final equilibrium it was only about 0.6%. Experiments have now been made in which a continuous current of dry air was passed through an apparatus in which it was subjected to the silent discharge. In the large scale apparatus used the discharge took place inside an aluminium tube 5 ft. long and 6 in. diam. In the axis of the tube was supported an aluminium rod  $\frac{1}{2}$  in. diam., and between this and the aluminium tube was a porcelain tube which served both as a high dielectric and to divide the space into two compartments. The air current passed first in one direction between the aluminium and porcelain tubes and then in the other between the porcelain tube and the rod. By means of a pump air could be passed through the apparatus at pressures above or below atmospheric. Trial runs were made at pressures of 460, 560, 660, 710, and 780 mm. at room temperature. At each pressure the production of nitric acid increased rapidly as the rate of passage of air increased, the maximum being attained at a rate of about 6 l. per min.; with greater velocities the yield diminished. With constant air velocity the yield increased rapidly with increasing pressure. The highest yield obtained was 9.8 g. HNO<sub>3</sub> per k.w.-hr. at 780 mm. and 6 l. per min., and the limit has certainly not been approached. At higher velocities the absorption of nitric acid was probably imperfect. In an analysis of the heat losses and energy consumed in producing nitric acid 25.8 watt-hours out of 134 are unaccounted for. On theoretical grounds, if all the electrical energy were available for the reaction, the yield would be 250 g. HNO<sub>3</sub> per k.w.-hr., whilst the arc process at 4200° C. can only be expected to give 134 g. per k.w.-hr. At equal efficiencies, therefore, the silent discharge process should give a higher yield and should be much cheaper.—E. H. R.

*Nitrogen pentoxide; Thermal decomposition of gaseous —.* F. Daniels and E. H. Johnston. *J. Amer. Chem. Soc.*, 1921, 43, 53–71.

THE thermal decomposition of nitrogen pentoxide is a unimolecular reaction at all temperatures from 0° C. to 65° C. The reaction has a large temperature coefficient, viz., 200–300% for 10°, and is not catalysed by the glass walls of the decomposition vessel. (Cf. J.C.S., April.)—J. F. S.

*Nitrogen pentoxide; Photochemical decomposition of —.* F. Daniels and E. H. Johnston. *J. Amer. Chem. Soc.*, 1921, 43, 72–81.

NITROGEN pentoxide is decomposed by light of wave length 400–460  $\mu$  if nitrogen dioxide is present. The autocatalytic effect of the dioxide is negligible in the dark. (Cf. J.C.S., April.)—J. F. S.

*Ammonia; Actual state of the synthesis of — at extremely high pressures.* G. Claude. *Comptes rend.*, 1921, 172, 442–444.

IN the latest development of the synthesis of ammonia at extremely high pressures (*cf.* J., 1919, 885 A; 1920, 61 A, 187 A, 655 A, 746 A) the compression of the reacting gases from 100 atm. to 900 atm. is effected in one stage in a hyper-compressor capable of dealing with 700 cb. m. of the gases per hr., a quantity sufficient for an apparatus with a capacity of 5 tons of ammonia per day. The contact unit now used comprises four tubes, two in parallel and then two in series. The heat of the reaction is carried away from the contact unit by a stream of molten lead.—W. G.

*Carbon monoxide; Absorption of — by cuprous ammonium carbonate solutions.* W. R. Hainsworth and E. Y. Titus. *J. Amer. Chem. Soc.*, 1921, 43, 1–11.

FOR the removal of the last portions of carbon monoxide from gas mixtures used in synthetic ammonia processes (after removing the major portion by oxidation to carbon dioxide and absorption of the latter in water), ammoniacal cuprous chloride solutions are unsatisfactory owing to the tendency to deposit copper and to gradual solution of the iron walls of the containing vessel. These tendencies are greatly reduced if an ammoniacal cuprous salt of an organic acid, *e.g.*, the formate or carbonate, is used instead of the chloride. The absorption capacity of a solution of cuprous ammonium carbonate for carbon monoxide is dependent on the concentration of cuprous copper, the partial pressure of the carbon monoxide, and the concentration of free ammonia. Absorption is due to the formation of an unstable compound,  $\text{Cu}_2(\text{NH}_3)_n\text{CO}_3 \cdot 2\text{CO} \cdot 4\text{H}_2\text{O}$ . The presence of small quantities of oxygen prevents the reduction of the cuprous copper to metallic copper and increases the absorption capacity of the solution. The solution of cuprous ammonium carbonate may be regenerated after use by heating to 75° C. in an inert atmosphere at ordinary pressure. (*Cf.* J. C. S., April.) —J. F. S.

*Trithiocarbonates and perthiocarbonates.* E. W. Yeoman. *Chem. Soc. Trans.*, 1921, 119, 38–54.

TWO well-defined series of thiocarbonates, derived respectively from trithiocarbonic acid,  $\text{H}_2\text{CS}_3$ , and perthiocarbonic acid,  $\text{H}_2\text{CS}_4$ , have been prepared, the methods adopted in general being the addition of the theoretical quantity of carbon bisulphide to an alcoholic or aqueous solution of the metallic hydrosulphide for the trithiocarbonates, or to a solution of the disulphide of the metal for the perthiocarbonates. From the hot alcoholic or aqueous solutions the salts were deposited in crystalline form on cooling or on addition of ether. The trithiocarbonates of sodium, potassium, ammonium, barium, strontium, and magnesium form yellow or red crystals, soluble in water, giving yellowish-red solutions. They are stable in dry air free from carbon dioxide, but are very hygroscopic, particularly the alkali trithiocarbonates, and if allowed to become moist they quickly oxidise and decompose with loss of carbon bisulphide. Aqueous solutions of the trithiocarbonates dissolve and combine with an additional atom of sulphur with formation of the perthiocarbonates, which are, however, more readily obtained by the alternative method above. The perthiocarbonates of the alkali and alkaline-earth metals are yellow, deliquescent, crystalline solids, soluble in water giving yellow solutions, from which mineral acids precipitate perthiocarbonic acid as a red oil. The calcium thiocarbonates all appear to be basic salts, which are more stable than the normal salts, and less hygroscopic.—G. F. M.

*Manganese salts; Reaction of —.* V. Macri. *Ann. Chim. Analyt.*, 1921, 3, 55–56.

THE author claims priority (J., 1917, 1176) for the oxalate reaction of manganese salts described by Caron and Raquet (J., 1919, 574 A).—W. P. S.

*Salts; Double decompositions of — and their geometric representation.* H. Le Chatelier. *Comptes rend.*, 1921, 172, 345–350.

A GRAPHICAL method for the investigation of systems composed of mixtures of salts is described. The method is illustrated by reference to the double decomposition,  $\text{NaNO}_3 + \text{NH}_4\text{Cl} = \text{NaCl} + \text{NH}_4\text{NO}_3$ , and a diagram is figured and described which facilitates the solution of the following problems:—The determination of the composition of mixtures of three salts; the determination of the surface of saturation; control of the progress of crystallisation and determination of the quantity of salt deposited; determination of the conditions under which there is re-solution of one of the salts at the invariant point.—W. G.

*Saline solutions; Displacement of metals in —.* Barlot. *Comptes rend.*, 1921, 172, 378–381.

THE precipitation of one metal from a solution of one of its salts by a more electronegative metal may most readily be followed by soaking a filter paper in a solution of the salt, placing it on a glass plate, and then placing a fragment of the more electronegative metal on the paper. (*Cf.* J.C.S., April.) —W. G.

*Fractional precipitation [of copper and silver and of cobalt and nickel].* P. Jolibois, R. Bossuet, and Chevry. *Comptes rend.*, 1921, 172, 373–375.

USING the method previously described (J., 1920, 84 A, 107 A), it was found that on adding increasing quantities of sodium hydroxide solution to a solution of copper and silver nitrates the fractionation is perfect, no silver appearing in the precipitate until sufficient sodium hydroxide has been added to precipitate the whole of the copper. On adding sodium hydroxide to a solution of nickel and cobalt chlorides the fractionation is imperfect, both metals always appearing in the precipitate, although in the early stages the nickel predominates.—W. G.

*Magnesium carbonate; Crystalline —.* T. C. N. Broeksmit. *Pharm. Weekblad*, 1921, 58, 210–212.

THE amorphous precipitate obtained by adding sodium, potassium, or ammonium carbonate to magnesium sulphate solution becomes crystalline on standing. No double salt is formed. Exactly similar crystals are obtained with calcium salts. —S. I. L.

*Silicic acid gels.* R. Schwarz. *Kolloid-Zeits.*, 1921, 28, 77–81.

SILICIC acid gels dissolve in ammonia to form both a colloidal solution and a molecular disperse solution, that is ammonium silicate. On keeping such a solution the amount of ammonium silicate increases at the expense of the colloidal silicic acid.—J. F. S.

*Colloids; Role of protective — in catalysis [of hydrogen peroxide by colloidal platinum].* T. Iredale. *Chem. Soc. Trans.*, 1921, 119, 109–115.

THE inhibitive effect of protective colloids on the catalytic decomposition of hydrogen peroxide by colloidal platinum decreases in the order: gelatin, egg albumin, tragacanth, dextrin, gum arabic. With a strong protective colloid, such as gelatin, the inhibitive effect is noticeable at a dilution as great as 0.000005%. The inhibition is explained on the ground of selective adsorption resulting in a decreased concentration of hydrogen peroxide at the platinum surface and a consequent fall in the



value of the velocity constant. The reaction may be used to detect not only adsorption effects, but probably also changes in state of the protective colloid, as the most striking fact about the results with varying concentrations of gelatin, for example, was the gradual rise of the velocity constant with diminishing concentration down to 0.00005%, and the rapid rise on further dilution, which seemed to indicate that the initial dilution caused the larger gelatin ultramicros to split into smaller ones down to a certain limit, thus compensating for the decreased concentration, after which further dilution of the colloid caused a rapid rise in the velocity constant.—G. F. M.

*Ruthenium and osmium; Action of hydrochloric acid on the tetroxides of —.* H. Remy. J. prakt. Chem., 1920, 101, 341–352.

THE contradiction between the statements that concentrated hydrochloric acid reacts with osmium tetroxide at the ordinary temperature, and that no action occurs (J., 1919, 133 A), is shown to be due to differences in strength of acid employed. Acid of sp. gr. >1.160 reacts at the ordinary temperature, giving osmium tetrachloride; the same acid with ruthenium tetroxide reacts still more readily, forming the trichloride.—J. K.

*Cyanogen chloride; Preparation and some physical constants of —.* C. Mauguin and L. J. Simon. Ann. Chim., 1921, 15, 18–41.

AFTER an examination of different methods for the preparation of cyanogen chloride, the authors find that the best yield is obtained by the action of chlorine on hydrocyanic acid (cf. Price and Green, J., 1920, 98 T). For the pure chloride the b.p. is 12.5° C. at 755 mm.; m.p., –7° to –6° C.; D, 1.222; mean coefficient of expansion between 0° and 45° C., 0.0015.—W. G.

*Iodine; Action of — in the cold on different metals. Process for detecting the presence of chlorine in the atmosphere.* C. Matignon. Comptes rend., 1921, 172, 532–534.

IODINE placed on thin sheets of metals such as silver, copper, aluminium, tin, or zinc gradually converts the whole of the metal into the iodide. The action spreads outwards from the point of contact, the velocity varying with the metal. For the detection of chlorine in the air, a thin sheet of beaten silver, 1 cm. in diameter, covered with moist potassium iodide and forming part of an electric circuit, is exposed to the air. Any chlorine present decomposes the potassium iodide and the iodine liberated converts the silver into silver iodide. The electric circuit is thus broken and by a suitable arrangement an audible warning may be given.—W. G.

*Carbon; Combustion of — in the presence of oxides.* E. Berger and L. Delmas. Bull. Soc. Chim., 1921, 29, 68–77.

CERTAIN metallic oxides (CoO, CuO, PbO, Sb<sub>2</sub>O<sub>3</sub>, MoO<sub>3</sub>; also PhCrO<sub>3</sub>) when mixed with carbon to the extent of 4% of its weight facilitate the combustion of the carbon in air. Other oxides cause a diminution in the proportion of carbon monoxide produced in the combustion of the carbon; among the most active are those oxides which are known as good catalysts for various oxidation processes, e.g., CuO, MnO<sub>2</sub>, Ag<sub>2</sub>O.—W. G.

See also pages (A) 206, *Ammonia from shale* (Lomax and Remfrey). 221, *Nitric acid and copper* (Bagster). 233, *Ammonia recovery in sugar factories* (Silhavy). 241, *Chlorine and bromine* (Ludwig); *Hydroiodic acid* (Hendrixson). 242, *Sulphuric acid* (Pezzi).

# PATENTS.

*Nitric acid; Process of concentrating dilute —.*

H. Frischer. E.P. 137,834, 12.1.20. Conv., 31.8.16.

DILUTE nitric acid is vaporised by introducing it into a liquid of high boiling point, which is unaffected by it, such as phosphoric acid, sulphuric acid, or arsenic acid, the liquid being heated to a high temperature (140°–160° C.) and the nitric vapours conducted to a dephlegmator. Instead of causing the dilute nitric acid to pass direct into the vaporising apparatus, it may first be introduced into the dephlegmator, from which a part is drawn off as concentrated acid, and the remainder then vaporised by the heating liquid.—W. J. W.

*Hydrofluosilicic acid gas; Absorber for —.* K. F. Stahl, Assr. to General Chemical Co. U.S.P. 1,367,993, 8.2.21. Appl., 18.8.17.

THE absorber consists of a tower with perforated bottom, the whole constructed of wood with an acid-resistant coating and filled with broken acid-resistant material.—C. I.

*Phosphoric acid; Process for making —.* W. H. Allen. U.S.P. 1,368,379, 15.2.21. Appl., 17.6.20.

CRUSHED phosphate rock is mixed with sand and coke, and the mixture is strongly heated, phosphorus pentoxide being evolved.—W. J. W.

*Soda containing water of crystallisation; Process for producing — or mixtures thereof with other substances.* A. Welter. E.P. 136,341, 18.12.19. Conv., 23.3.18.

A FINELY divided steam jet, shower of snow, or spray of water or of a solution of the salt which it is desired to mix with the soda, e.g., water-glass, is directed on to soda ash which is kept in motion to prevent caking. The process may be carried out in a tower, or the spray may be used to blow the soda ash into a powder chamber, or the material may be sprayed on a conveyor belt provided with rakes, or in an agitator. In all cases cooling is necessary. A washing powder fit for sale is thus produced without further treatment.—C. I.

*Ammonium perchlorate; Manufacture of —.* L. M. E. Wang. E.P. 137,034, 21.11.19. Conv., 27.12.18.

TO an aqueous solution of ammonium sulphate and sodium perchlorate at a temperature below 100° C. but considerably above that of maximum solubility of sodium sulphate, further quantities of the reacting salts are added. The precipitated sodium sulphate is then separated from the solution, from which by cooling ammonium perchlorate is recovered, and the process is repeated. Alternatively, the hot mother liquor may be treated in separate portions with the additional quantities of sodium perchlorate and ammonium sulphate, after which the solutions are mixed.—W. J. W.

*Hydrogen sulphide; Production of — from sulphurous gases.* J. G. Macleod. From W. J. Browning. E.P. 158,288, 31.7.19.

SULPHUROUS gases formed by the combustion of pyrites etc. are passed through incandescent carbonaceous matter, the heat of which is maintained by its own combustion. A regulated quantity of oxygen and, if necessary, water vapour is mixed with the sulphurous gases.—C. I.

*Cyanides; Manufacture of —.* O. L. Barnehey. U.S.P. 1,355,642, 12.10.20. Appl., 19.3.19.

ACTIVATED carbon, sodium carbonate, and a catalyst, e.g., iron, are heated in an atmosphere of nitrogen. The activated carbon is prepared by heating carbon in presence of steam or carbon dioxide above 700° C.—C. I.

*Nitrates; Process for fixation of* —. C. H. Buettner. U.S.P. 1,368,019, 8.2.21. Appl., 6.3.19.

Nitrogen and oxygen are brought into contact with water in small quantities, and the mixture is subjected to the action of the electric arc.—W. J. W.

*Nitrogen compounds with hydrogen and oxygen; Preparation of* — by the use of a titanium catalyst. F. Hlavaty and F. J. G. Oehlbach. G.P. 323,945, 5.9.16. Addn. to 275,343.

EITHER spongy metallic titanium or finely divided titanium carbide is used as catalyst without any carrying medium.—C. I.

*Alkali-metal compounds; Process of obtaining water-soluble* — from water-insoluble substances. H. S. Blackmore, Assr. to Kali Co. of America. U.S.P. 1,357,025, 26.10.20. Appl., 14.12.18.

SILICATES insoluble in water and containing an alkali metal are heated under pressure with water and a non-acid metal silicofluoride, e.g., magnesium silicofluoride. After filtering and cooling, potassium silicofluoride, sparingly soluble in cold water, separates, and this is subsequently converted into more soluble salts, such as the carbonate, sulphate, etc.—C. I.

*Chlorides of potassium or other metals; Process for obtaining* —. W. A. Schmidt, Assr. to International Precipitation Co. U.S.P. 1,367,836, 8.2.21. Appl., 29.4.18.

POTASSIUM chloride is recovered from material containing alkali chlorides and sulphates by adding a chloride less volatile than potassium chloride. The mixture is heated and the whole of the potassium recovered as chloride vapour.—C. I.

*Potassium nitrate and mixtures of salts containing potassium and nitrogen; Manufacture of* —. Badische Anilin u. Soda Fabrik. G.P. 310,601, 8.11.20. Addn. to 306,334 (J., 1921, 179 A).

By cooling a hot solution of 98.5% ammonium nitrate, 204 pts.; 98.7% potassium chloride, 206 pts.; and water, 425 pts., to 2° C., 165 pts. of 99% potassium nitrate is recovered. On evaporating the mother liquor a mixture is obtained which contains about 36% of potassium nitrate, 55% of ammonium chloride, 8% of potassium chloride, and 1% of sodium chloride, and which has good keeping properties in a moist atmosphere.—W. J. W.

*Cooling apparatus for hot solutions of potassium salts etc.* H. Daus. G.P. 329,479, 15.2.20.

THE solution is forced through a nozzle giving a jet in the form of a hollow cone, while cooling air is sucked or driven up the centre of this cone and through the solution.—C. I.

*Thorium nitrate; Process of manufacturing* —. C. W. Davis, Assr. to The Mantle Lamp Co. of America, Inc. U.S.P. 168,243, 15.2.21. Appl., 16.9.18.

A RARE earth metal, e.g., thorium, is separated from a mixture containing it by treatment with an alkali carbonate in presence of a phosphate.—C. I.

*Inorganic materials [containing iron]; Process of purifying* —. G. A. Hulett. U.S.P. 1,368,396, 15.2.21. Appl., 22.3.19.

IRON and other impurities which form volatile chlorides are removed by the action of a current of phosgene at 350°–600° C.—C. I.

*Bleaching powder; Process and apparatus for the manufacture of* —. E. Sidler. G.P. 329,178, 13.12.17.

THE chlorinating chamber contains a series of superimposed plates and stirring arms, which continuously turn over the lime and rake it from the

sides towards the middle on one plate and from the middle towards the sides on the next lower one and so on. The lime is charged on to the top plate, and is gradually raked down from one plate to the next to the bottom of the chamber, the chlorine being passed in the same direction.—A. R. P.

*Ammonium sulphate; Preparation of powdered non-caking* —. A.-G. für Anilinfabr. G.P. 329,359, 27.5.19.

SOLUTIONS of ammonium bisulphate or sulphuric acid are sprayed through ammonia gas under pressure.—C. I.

*Hydrogen and oxygen; Catalytic purification of* —. Consortium für Elektrochem. Ind. G.P. 329,177, 17.10.19.

THE gases are passed over heated mixtures of copper and silver, or of their compounds, with or without compounds of silicon, cerium, and zirconium. The porcelain masses carrying the catalyst require to be heated to 200°–350° C., according to the composition of the latter.—C. I.

*Sodium-ammonium sulphate; Separation of the double salt* — into sodium sulphate and ammonium sulphate. Separation of sodium sulphate and ammonium sulphate from the double salt sodium ammonium sulphate. Soc. Ind. de Prod. Chim. E.P. 128,895, 27.9.18 (Conv., 22.6.18), and 158,282, 6.1.19. Addns. to 136,833 (J., 1920, 516 A).

SEE U.S.P. 1,364,822 of 1921; J., 1921, 147 A.

*Ammonia solution; Method of treating crude gas liquor to obtain* —. C. Still. E.P. 147,099, 7.7.20. Conv., 6.12.15.

SEE G.P. 302,195 of 1915; J., 1918, 334 A.

*Sodium permanganate; Manufacture of* —. D. Tyrer. U.S.P. 1,368,076, 8.2.21. Appl., 29.9.19.

SEE E.P. 130,844 of 1918; J., 1919, 719 A.

ERRATUM.—This J., Mar. 15, 1921, p. 146 A, col. 2, line 7 from bottom, for "for raising the efficiency of" read "of making" — C. J.

See also pages (A) 204, *Interaction of liquid or pulverulent materials* (G.P. 302,421). 205, *Recovering sulphur dioxide and ammonia* (G.P. 329,412). 225, *Electrolytic gas generators* (E.P. 158,148). 233, *Alunite ore* (E.P. 158,293). 240, *Potassium chlorate* (U.S.P. 1,355,203).

## VIII.—GLASS; CERAMICS.

*Glass; Development of various types of* —. C. J. Peddle. J. Soc. Glass Tech., 1920, 4, 299–366.

THE preparation, stability, and optical properties of the following series of alkali, lead oxide, silica glasses were studied:—100 SiO<sub>2</sub>, 40 Na<sub>2</sub>O, x PbO; 100 SiO<sub>2</sub>, 20 Na<sub>2</sub>O, x PbO; 100 SiO<sub>2</sub>, 40 K<sub>2</sub>O, x PbO; 100 SiO<sub>2</sub>, 20 K<sub>2</sub>O, x PbO; 100 SiO<sub>2</sub>, 20 Na<sub>2</sub>O, 20 K<sub>2</sub>O, x PbO; 100 SiO<sub>2</sub>, 10 Na<sub>2</sub>O, 10 K<sub>2</sub>O, x PbO, where x varied from 5 to 40 mols. All the glasses can be founded above 1350° C. and exhibit corrosive action on pot clay, increasing with increased alkali content. Glasses of the soda series tend to devitrify if containing more than 65% SiO<sub>2</sub>, and those of the mixed alkali series if the silica content exceeds 61%. The potash glasses show no signs of devitrification, but all film badly on prolonged heating at 900° C. In all cases density and refractive index increase with increasing content of lead oxide, the rate of increase falling as the lead oxide content rises. Dispersion also increases with increase of lead oxide, but  $v$  falls as  $n_D$  rises. Comparison of the above and also of further series containing equal percentage amounts of potash and soda

showed all the soda glasses to have higher densities than the corresponding potash glasses, the mixed alkali glasses having intermediate values. In the equimolecular series the potash glasses have slightly higher refractive index and total dispersion, but in the series containing equal weights of alkali the soda series have higher refractive index and dispersion. The refractive index and dispersion of the mixed alkali series are intermediate between those of the soda and the potash series. The solubility of the glasses in water decreases as the content of lead oxide or silica rises but increases rapidly with increase of alkali. Solubility tests prove the following glasses to be of first-class durability for optical requirements:—Glasses of the molecular formulae,  $20 \text{ Na}_2\text{O}$ ,  $30 \text{ PbO}$ ,  $100 \text{ SiO}_2$ ;  $20 \text{ Na}_2\text{O}$ ,  $40 \text{ PbO}$ ,  $100 \text{ SiO}_2$ ;  $10 \text{ Na}_2\text{O}$ ,  $10 \text{ K}_2\text{O}$ ,  $30 \text{ PbO}$ ,  $100 \text{ SiO}_2$ ;  $10 \text{ Na}_2\text{O}$ ,  $10 \text{ K}_2\text{O}$ ,  $40 \text{ PbO}$ ,  $100 \text{ SiO}_2$ ;  $20 \text{ K}_2\text{O}$ ,  $40 \text{ PbO}$ ,  $100 \text{ SiO}_2$ ; also glasses of the percentage compositions,  $\text{SiO}_2$  50,  $\text{Na}_2\text{O}$  10,  $\text{PbO}$  40;  $\text{SiO}_2$  50,  $\text{K}_2\text{O}$  10,  $\text{PbO}$  40;  $\text{SiO}_2$  40,  $\text{Na}_2\text{O}$  10,  $\text{PbO}$  50;  $\text{SiO}_2$  40,  $\text{K}_2\text{O}$  10,  $\text{PbO}$  50;  $\text{SiO}_2$  60;  $\text{Na}_2\text{O}$  5,  $\text{K}_2\text{O}$  5,  $\text{PbO}$  30;  $\text{SiO}_2$  50,  $\text{Na}_2\text{O}$  5,  $\text{K}_2\text{O}$  5,  $\text{PbO}$  40;  $\text{SiO}_2$  40,  $\text{Na}_2\text{O}$  5,  $\text{K}_2\text{O}$  5,  $\text{PbO}$  50. The first two of the molecular series approximate in optical constants to well-known Schott glasses. The Benrath formula  $6 \text{ SiO}_2$ ,  $1 \text{ R}_2\text{O}$ ,  $1 \text{ PbO}$  does not give a very stable type of alkali-lead oxide-silica glass, a more satisfactory one being  $5 \text{ SiO}_2$ ,  $1 \text{ R}_2\text{O}$ ,  $1.5 \text{ PbO}$ .—A. C.

*Glass composition; Rate of change of — following a change in batch composition.* J. Currie. J. Soc. Glass Tech., 1920, 4, 382—387.

On changing the composition of a lime-soda batch supplying a tank furnace so as to increase the lime content of the glass produced, the relationship between capacity of tank, rate of feeding in the fresh batch, and the time taken for the glass worked out to approximate to the desired new composition, was noted. The furnace capacity was 106 tons, the weekly addition of batch 95 to 110 tons (containing only about one-eighth of added cullet from the working end). The change of composition was not quite complete in 3 months.—A. C.

*Glass; Influence of silica on the annealing temperature of —.* S. English, F. W. Hodkin, C. M. M. Muirhead, and W. E. S. Turner. J. Soc. Glass Tech., 1920, 4, 337—391.

A SERIES of simple sodium silicate glasses ranging from  $2\text{Na}_2\text{O} \cdot 4\text{SiO}_2$  to  $2\text{Na}_2\text{O} \cdot 10\text{SiO}_2$  was examined. The upper annealing temperature was found to rise with increase of silica content, the rate of increase being most rapid between  $2\text{Na}_2\text{O} \cdot 4\text{SiO}_2$  and  $2\text{Na}_2\text{O} \cdot 7\text{SiO}_2$ .—A. C.

*Glass; Devitrification of — as a surface phenomenon. Repair of crystallised glass apparatus.* A. F. O. Germann. J. Amer. Chem. Soc., 1921, 43, 11—14.

DEVITRIFICATION of glass in a blowpipe flame is not due to the formation of crystal nuclei in the mass of the glass but is a surface phenomenon due to the expulsion of water and carbon dioxide, and possibly, when the heating is prolonged, to the volatilisation of sodium oxide. Devitrification may be prevented by washing the parts to be heated with dilute hydrofluoric acid, thus removing the surface layer. In this way old apparatus which has been broken may be repaired.—J. F. S.

*Clay; Ageing of —.* H. Spurrer. J. Amer. Ceram. Soc., 1921, 4, 113—118.

The gases recovered by the method described previously (J., 1919, 176 A, 633 A) from clay that had been pugged 24 hrs. previously were found to contain both carbon monoxide and carbon dioxide. The evolution of carbon dioxide continued for over 34 days after pugging and, like the change of plas-

ticity, proceeded more rapidly between  $80^\circ$  and  $90^\circ \text{ F.}$  than below  $60^\circ \text{ F.}$  By replacing water by non-aqueous liquids the development of plasticity was inhibited entirely, suggesting that hydroxyl ions must be present in order to develop plasticity. The addition of a dilute solution of hydrogen peroxide to the clay slip produced a pronounced increase of viscosity and stimulated the growth of filamentous algae with the consequent evolution of both carbon monoxide and carbon dioxide. The change of plasticity of clays with time seems due, in some way, to the growth of such algae, though the copious formation of carbon dioxide is somewhat confusing, because the introduction of free carbon dioxide reduces the plasticity of clays. The ratio of the amounts of alumina and silica dissolved by caustic potash was found for the three clays tested to decrease rapidly with diminishing plasticity and might therefore be used as a quantitative measure of plasticity.—H. S. H.

*Aluminous fireclays; Effect of added felspar on the shrinkage and porosity of — after being fired at high temperature.* E. M. Firth and W. E. S. Turner. J. Soc. Glass Tech., 1920, 4, 392—400.

IN a previous paper (J., 1920, 628 A) the authors had noted that, of clays which they examined, those of high alumina content showed, with two exceptions, the widest range of porosity on firing. It is now shown that if 1 or 2% of felspar is added to the two exceptional aluminous clays (to act as a flux during firing) the shrinkage is rendered more uniform, and there is a considerable increase of porosity range.—A. C.

*Spalling [of bricks]; Study of —.* R. M. Howe and R. F. Ferguson. J. Amer. Ceram. Soc., 1921, 4, 32—46.

BRICKS were heated in the door of a furnace and then rapidly cooled, some by standing in water to a depth of 5 in. and others by directing a jet of cold air from a blower on them. In each case the loss in weight due to the detachment of particles was taken as a measure of the amount of spalling. Both spalling tests gave similar results. With bricks made from the same clay, lightly burned bricks spalled less than hard burned bricks, and resistance to spalling was increased by using coarser ground material. These tests lead to accurate conclusions where the same fireclays are involved, but misrepresent the facts observed in works service when different clays are concerned. These discrepancies disappeared when the specimens were heated for 5 hrs. at  $1400^\circ \text{ C.}$  before testing. Two fireclays which produced bricks which were very resistant to spalling when in use were characterised by a constancy in porosity over a wide range of temperature, and were therefore considered very resistant to vitrification. When firebricks spall in service the pieces are usually dense or vitrified. The connexion between vitrification and spalling was established by experiments. Fireclays which undergo a slight change in porosity when heated over a wide range of temperature have a very slight tendency to vitrify, and are particularly adapted to the manufacture of non-spalling bricks.—H. S. H.

*Spalling [of firebrick]; Use of plastic clay grog in preventing —.* R. M. Howe and S. M. Phelps. J. Amer. Ceram. Soc., 1921, 4, 119—126.

A MEDIUM dense burning clay was calcined at cone 8, then ground and mixed with plastic clay in various proportions up to 40%, and the bricks made therefrom were burned at cone 8. As the percentage of grog increased the bricks became more porous and mechanically weaker, but showed less drying and burning shrinkage. The water spalling test was too severe for this type of brick, and the difference in the results obtained was too slight to permit

definite conclusions being drawn. An air-spalling test of the bricks showed that the grog increased the resistance to spalling about 5% for each 1% of grog added.—H. S. H.

*Silica brick; Note on —.* D. W. Ross. J. Amer. Ceram. Soc., 1921, 4, 65—67.

OBSERVATIONS were made during an extended period on the silica bricks in the crown of a tank furnace used in the manufacture of bottle glass. It was found that great care was necessary in heating up a new crown, due both to the large expansion of the whole crown and to unequal expansion from place to place, some portions expanding considerably more than others. After having been in use for 18 months (or more) a crown could usually be cooled without disruption if the temperature did not drop too suddenly after the fires were turned off. On reheating the crowns only expanded one-half as much as they did in the first heating, and local non-uniformity was practically absent. When a crown had been cooled after being in use for 18 months a large percentage of the bricks were broken in two or contained a source of weakness approximately midway between the inside and outside of the crown. Microscopical examination of the bricks after 18 months' service indicated that they were practically all tridymite, whereas the unused bricks were largely cristobalite and quartz.—H. S. H.

*Dolomite and magnesite; Experiments in dead-burning —.* H. G. Schurecht. J. Amer. Ceram. Soc., 1921, 4, 127—151.

DOLomite is difficult to dead-burn owing to its tendency to disintegrate, due partly to the inversion of  $\beta$ -to- $\gamma$ -calcium orthosilicate on cooling, with a 10% volume increase, and partly to gradual slaking owing to the hydration of free lime. With all the fluxes tried (flue dust, iron ore, basic open-hearth slag, kaolin, roll-scale) dolomite passed through a 100-mesh sieve gave greater resistance to slaking than when sieved through 8-mesh. This resistance decreased with increasing temperature of calcination by an amount which seemed to depend on the silica and alumina content of the flux, being greatest for basic open-hearth slag and kaolin, less for flue dust and iron ore, and least for roll-scale. This result is possibly due to the formation of hydraulic compounds with lime, similar to those found in Portland cement. The stability also varied with the proportions of flux used, being greater, as a rule, for smaller proportions. The magnesite used contained about 11% of lime, and increasing the calcination temperature decreased the tendency of the product to increase in weight on exposure to the atmosphere. Roll-scale was a more effective flux for magnesite than was iron ore, probably because of its lower silica content. The shrinkage and porosity curves for mixtures of dolomite with flue dust, iron ore, and roll-scale respectively were very similar. The higher the proportion of flux, the lower was the temperature required to obtain a product of maximum shrinkage and minimum porosity. Kaolin and basic open-hearth slag behaved peculiarly in that vitrification depended more on the burning temperature than on the proportion of flux used. Kaolin was more effective than an equal weight of iron ore or flue dust in promoting vitrification. With magnesite and roll-scale the shrinkage was much less and the porosity much greater than with dolomite and roll-scale, the lime of the dolomite being more actively attacked by roll-scale than was magnesite. Mixtures of dolomite and iron ore were passed through a 100-mesh sieve, calcined to cone 20, and moulded into bricks with 5% of caustic lime, sodium silicate, or magnesium chloride. Owing to slaking all the bricks cracked on drying. This was avoided by "ageing" the batches with excess of water for at

least three weeks. Hydration had then taken place and no cracking occurred on drying. Bricks made with magnesium chloride were then very strong. On burning to cone 18 all cracked excessively, due to high shrinkage, but withstood storage for 4 to 6 months. Bricks pressed from calcined dolomite with 12% of dehydrated coal tar withstood storage for about 4 months. They showed less shrinkage and cracking on firing than similar bricks made with water, and if dipped in tar to protect them from moisture, could be stored for 5—6 months.

—H. S. H.

*Terra-cotta; Effect of glaze composition on the crazing of —.* E. C. Hill. J. Amer. Ceram. Soc., 1921, 4, 25—31.

THE body used contained 35% of grog and was composed of equal parts of a fat plastic clay which had shown a tendency to produce crazing and a clay which might be considered medium in this respect. The test pieces were pressed from this body, and when dry sprayed with the engobe regularly used, and over this was placed a heavy coat of glaze. The specimens were fired to cone 5 $\frac{1}{2}$  in 40 hrs. and cooled in about the same time; two burns were made. The glaze used as a base had the composition (0.28 K<sub>2</sub>O, 0.21 CaO, 0.21 ZnO, 0.10 MgO, 0.20 BaO); (0.33 Al<sub>2</sub>O<sub>3</sub>), (2.53 SiO<sub>2</sub>, 0.10 SnO<sub>2</sub>). Comparing the effects of equal molecular additions of the various components of the glazes, it was found that zinc oxide and felspar overcame crazing, the former being the more effective. Magnesia, barium monoxide, and lime tended to produce crazing, magnesia having the greatest tendency, and the effect of barium monoxide being somewhat greater than that of lime. Tin oxide had no effect on the crazing. Additions of small amounts of clay were more effective in overcoming crazing than much larger additions or substitutions of the other components. Increase of flint (per molecular equivalent) was less effective in overcoming crazing than increase of either clay, zinc oxide, or felspar, although, generally, considerably more flint than clay or zinc oxide could be added without appreciably changing the maturing of the glaze. The results indicated that in many cases increase of flint will not overcome crazing in a glaze.—H. S. H.

#### PATENTS.

*Sheet glass; Process and apparatus for the manufacture of —.* R. L. Frink. E.P. 157,598, 6.11.19.

A RECTILINEAR metallic bait having a coefficient of expansion different from that of glass is lowered into a bath of molten glass and then raised. An electric current is passed through the bait, and is so regulated that the temperature of the bait is such that the glass in contact with it is kept from setting, and is thus able to accommodate itself without rupture to any relative displacement of the bait due to a change in temperature. The bait is raised at such a speed that a sheet of glass of substantially uniform thickness is produced. The edges of the sheet adjacent to the bath are kept in adhesive contact with electrically heated "shoes." The tendency of the glass to draw in at the sides owing to surface tension is thus counteracted, and the width of the sheet is maintained substantially constant. The sheet is severed from the bath, and the bait and the attached sheet of glass are moved in a horizontal direction out of alignment with the bath. Successive portions of desired length are then cut from the lower end of the sheet.—H. S. H.

*Glass melting tanks or furnaces.* C. Truel. E.P. 157,684, 15.6.20.

THE flues of a glass melting furnace are placed at the same end of the furnace and at about the same

level as the burners for liquid or gaseous fuel. The flames or products of combustion pass across the furnace to the opposite end, and are then brought back over the entire length of the furnace by the draught of the flues to the same end as that from which they were originally projected.—H. S. H.

*Quartz glass; Process of producing articles of —.*  
J. Scharl, Assr. to General Ceramics Co. U.S.P.  
1,368,990, 15.2.21. Appl., 18.9.18.

ARTICLES of quartz glass are made by heating plastic quartz in an electric resistance furnace about a resistance core, a space between the quartz and the resistance core being formed by inserting a heat-resisting tube, impervious to gas, to allow the escape of the gas formed during the heating.  
—A. B. S.

*Tunnel furnaces, kilns, ovens and the like.* H. Francart. E.P. (a) 157,474 and (b) 157,475, 19.12.18.

(A) In the interior of a tunnel furnace are a number of hollow flanges which serve as a part or the whole of the combustion chambers or hot gas flues. The hollow flanges are arranged in series so as to leave between them a series of vertical cavities along the oven with a view to favouring both radiation and efficient working. The flanges are provided with pipes traversing them from one vertical cavity to another in order to increase the circulation of the gaseous medium and to increase the heating surface. The pipes may be inclined so that the heated gas as it rises in the pipes also moves forward in a longitudinal direction. (b) A tunnel furnace has a number of solid flanges projecting inwards from the fixed walls so that vertical cavities are formed between the flanges. Spaces for combustion are provided at the bottom of the cavities. The flanges may be provided with suitable projections so as to increase the surface of radiation. Conduits are arranged at the bottom of the flanges so as to favour the longitudinal displacement of the gaseous mass, and this is also facilitated by inclining the flanges from the bottom upwards towards the entrance of the oven. By extending the flanges below the level of the sole of the furnace an efficient circulation is obtained in a low oven such as is used in annealing steel.—H. S. H.

*Kilns of the tunnel type; Continuous —.* R. C. Metcalfe. E.P. 158,499, 16.9.20.

A CONTINUOUS tunnel kiln is built in sections each at an angle to the preceding one, and is provided with turntables and pushing and engaging devices to facilitate the transference of the cars from one section to another.—A. B. S.

*Tunnel-kiln.* P. d'H. Dressler, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. 1,366,369, 25.1.21. Appl., 17.12.19.

A TUNNEL-KILN having an elongated kiln chamber has a heating zone and a goods cooling zone at one end of the heating zone. An air preheating and goods cooling conduit extends along the cooling zone, and a combustion chamber, separated by a heat-conducting wall from the kiln chamber, extends along the heating zone. A fan or blower having its inlet connected with the cooling zone and its outlet connected with the combustion chamber minimises the pressure differences tending to cause leakage flow between the kiln chambers and the combustion chamber or conduit.—H. S. H.

*Tunnel-kiln.* A. C. Shepard, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. 1,366,424, 25.1.21. Appl., 26.1.20.

A TUNNEL-KILN has masonry benches extending longitudinally at the sides of a pathway at the bottom of the kiln for the trucks carrying the ware. On each side of the benches is an elongated com-

bustion chamber from which extend pipes connected at their other end with a "header." Each bench is formed with a channel having uprising end portions which communicate one with the interior of the "header" and the other with the interior of the combustion chamber at the opposite ends of the pipes, and also with an outlet from the channel for the products of combustion. Dampers regulate the relative amounts of the products of combustion which pass from the combustion chamber by means of the pipes and a portion of the channel to this outlet and through the by-pass about the pipes formed by the remaining portion of the channel.—H. S. H.

*Kilns; Arrangement for utilising the waste gases in —.* F. Mayer. G.P. 328,985, 7.9.15.

WATER-PIPES are fixed in heating flues in the crown of the kiln, the flues being divided into cells which are of the same length as the burning chamber beneath and are in open connexion with the corresponding combustion chamber.—A. B. S.

*Brick-burning; Apparatus and method for —.*  
H. O. Robinson and C. Steadman. U.S.P.  
1,367,756, 8.2.21. Appl., 30.12.13.

THE bricks in a kiln are burned by a method comprising the following stages:—Using a low fire and forcing the heat by artificial draught throughout the entire stack; increasing the heat to full fire for 24–36 hrs.; forcing the heat in the lower courses of bricks upwards through the upper courses by means of artificial draught; continuing the heating until the lower courses of bricks are fully burned; and completing the burning of the upper courses by blowing the heat from the lower part into the upper part of the kiln.—A. B. S.

*Joints for earthenware or ceramic materials.* W. Hayhurst. E.P. 158,427, 24.11.19.

EARTHENWARE or other ceramic articles may be joined together or to metal by filling a recess in the ceramic article with a cement composed of a phenolic aldehyde condensation product combined with about one-third of a suitable filling material such as asbestos. The cement is hardened by heating it to 100°–150° F. (38°–65° C.) for 2–3 hrs., and is then tapped, threaded, or otherwise machined in order that it may be attached to the metal or other article.—A. B. S.

*Glass; Method and apparatus for forming sheet —.* E. Dauner. E.P. 159,114, 26.5.20.

## IX.—BUILDING MATERIALS.

*Plaster; Normal consistency of sanded gypsum —.*  
W. E. Emley and C. F. Faxton. J. Amer. Ceram. Soc. 1921, 4, 152–157.

TWELVE plasters were prepared containing various proportions of gypsum and sand, and to samples of each five different amounts of water were added. The consistencies, determined with a Southard viscosimeter (Amer. Soc. Testing Materials No. C 26—19T, 1919), and the tensile strengths of the set plasters, were tabulated. The results varied both with the kind of gypsum and with the fineness of the sand, but the opinion is expressed that material of the proper consistency to be used for plastering would show a "slump" of  $\frac{1}{4}$  in. when tested in the Southard viscosimeter.—H. S. H.

### PATENTS.

*Asphaltic cement or bituminous composition for road-making, paving, flooring, roofing, and other purposes.* M. Talbot-Crosbie. E.P. 158,674, 7.11.19.

AN asphaltic cement is formed by mixing heated bitumen or asphalt (45%) with purified chalk (55%).

A small quantity of linseed oil or other vegetable oil may be added to improve the weather-resisting properties. Sand may be mixed with the composition before use.—A. B. S.

*Silicated cement; Process of manufacturing* — S. Matsuo. U.S.P. 1,367,984, 8.2.21. Appl., 9.8.17.

Hot Portland cement clinker is added to wet sand which has been washed in hydrochloric acid, and the mixture is cooled and ground.—A. B. S.

*Shaft-furnace for cement and similar material*. A. Barth. G.P. 323,319, 20.5.19.

In a shaft-furnace of the type in which the grate is fixed and the shaft rotates, the lower part of the shaft is provided with arms arranged like the spokes of a wheel. Cold air is circulated through the arms and through passages in the casing enclosing the shaft. The arms are fixed to the shaft and hence rotate above the grate, and serve to promote uniform distribution of the material and to prevent agglomeration of large lumps.—W. J. W.

*Shaft kilns for burning lime, magnesite, cement, etc.; Device for preheating the air used for combustion in* — G. Polysius. G.P. 328,320, 12.2.18.

The air is drawn downwards through an annular space formed by iron rings around the cooling zone, and is thereby heated before it enters the lower end of the kiln.—A. B. S.

*Marl; Method of burning* — J. Mühlen. G.P. 329,167, 26.2.18.

The portion of the calcined marl which can only be slaked with difficulty is finely ground, mixed with the portion which slakes readily, and the mixture is slaked under such conditions that a dry powder is produced.—A. B. S.

*Waterproof kieselguhr; Production of* — W. Bunte and W. North. G.P. 329,410, 24.11.18.

Granulated or powdered kieselguhr is mixed with lightly ground pitch and the mixture is heated. The product is used as an insulating agent against heat, cold, moisture, and noise.—A. B. S.

*Cement; Slow-setting — and method of producing same*. H. D. Baylor. E.P. 153,390, 8.11.19.

See U.S.P. 1,323,952 of 1919; J., 1920, 113 A.

*Wood-like material*. U.S.P. 1,357,647. See V.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*High frequency induction steel furnace*. E. F. Northrup. Chem. and Met. Eng., 1921, 29, 309—311.

The Ajax-Northrup high frequency induction furnace operates with a current of from 10,000 to 25,000 cycles per sec., obtained through a suitable high frequency converter. The furnace consists essentially of an inductor coil in the shape of a truncated cone, which fits loosely round a crucible of similar shape. The coil itself is composed of 37 turns of  $\frac{3}{8}$ -in. copper tubing, flattened, and wound edgewise. During the heating process water is circulated through the coil to keep the resistance low. The inside of the inductor coil is lined with a cylinder of micanite about  $\frac{1}{4}$  in. thick, and a space of about  $\frac{1}{4}$  in. between the crucible and the coil is filled with a suitable powdered heat insulator, e.g., lampblack or powdered magnesia. The whole is encased in a box of stout asbestos board. As the heating of the crucible contents is due entirely to the large induced currents, the metal to be melted

must not be in the form of powder. Turnings, or pea-sized material, offer the most suitable form for efficient use. If the metal is magnetic there is a further development of heat in the earlier stages due to the hysteresis losses caused by the rapid reversals of the magnetism, and as soon as the metal becomes molten, an active circulation, due to electromagnetic forces, is set up, thus effecting thorough mixing of the melt. No conclusive tests on the maximum temperature attainable have been made, but 1800° C. is given as a conservative figure. Such metals as brass, copper, gold, and silver have too high conductivity to be melted efficiently by direct induction, and for this class of metals the non-conducting crucible is replaced by a crucible moulded from carbon or graphite. In this case the heat is generated directly in the walls of the crucible and is transmitted to the metal by conductivity and radiation.—C. A. K.

*Case-hardening; Nitrogen and* — H. Fay. Chem. and Met. Eng., 1921, 24, 289—290.

EXPERIMENTAL figures are given in support of the view that case-hardening is a nitrogenising as well as a carbonising process. Hurum found that after heating a low carbon steel in a cyanide bath for 10 mins. at 830° C., the outside layer contained 0.57% N. Nitrogen appears to penetrate at a slightly greater rate than carbon. Results obtained by treating steel by Shimer's cyanamide process (U.S.P. 1,279,457—8; J., 1918, 772 A) show that diffusion of carbon takes place below A<sub>1</sub> (at 650° C.), and that nitrogen combines with iron and diffuses below the A<sub>1</sub> point. The nitrogen content decreases with rise of temperature, indicating that equilibrium is reached at some lower temperature. In these experiments the maximum nitrogen content (0.579%) was attained at 750° C.—C. A. K.

*Steel welds; "Blue brittleness" of* — C. Frémont. Comptes rend., 1921, 172, 368—370.

By electrical welding under pressure sufficient to cause all the metal which is oxidised at the beginning of the operation to flow out laterally, a joint is obtained free from oxidised metal. In some cases this gives a satisfactory joint, but in other cases there is brittleness due to deformation within a critical temperature range. At a short distance from the joint, a region, for which the temperature is between 200° and 450° C., undergoes deformation under the action of the pressure necessary for the welding, and "blue brittleness" results. This would be avoided by choosing a steel which is not subject to blue brittleness (cf. J., 1905, 31).—W. G.

*Corrosion of steel ranges*. O. W. Storey. Trans. Amer. Electrochem. Soc., 1921, 13—21. [Advance copy.]

THE metal walls forming the flues of a domestic range may suffer corrosion, either from direct oxidation due to excessive heating, or, more extensively, from the deposition of moisture and sulphurous and other acids during the periods of warming or cooling the flues. Protective coatings of vitreous enamel or magnetic oxide would prevent corrosion. Steels containing copper are much less corroded than plain carbon steels or "Armco" iron, the resistance of copper steels (containing up to over 2% Cu) to corrosion being shown as a straight line function of the copper content.—C. A. K.

*Nickel steels; Electrical resistance of* — A. Portevin. Comptes rend., 1921, 172, 445—447.

THE electrical resistance of nickel steels is dependent to a considerable extent on the conditions of annealing. With steels containing 0.3—0.8% C and 7—15% Ni the resistance varied 10—40% according as the steel was heated to 1000° C. and cooled during 4—5 hrs., or heated to 1300° C. and cooled



during 3 days. This variation in the electrical resistance may or may not be accompanied by characteristic modifications of the micro-structure. Curves are given showing the variation in resistance of nickel steels according to their carbon and nickel content.—W. G.

*Nitric acid and copper; Reaction products of —.*  
L. S. Bagster. Chem. Soc. Trans., 1921, 119, 82–87.

THE gaseous products from the reaction of copper with 5N and 6N nitric acid consist almost entirely of nitrogen trioxide. With 14.5N acid, about 85–90% of nitrogen peroxide is formed, the residue being trioxide. From 10N acid, 15–20% of peroxide is produced; and from 8N acid, a slightly smaller amount.—W. J. W.

*Copper; Action of reducing gases on heated —.*  
H. Moore and S. Beckinsale. Inst. Metals, March, 1921. [Advance copy.] 22 pages.

SAMPLES of copper containing 0.08–0.02% of oxygen, and others of deoxidised copper, were subjected to the action of hydrogen, coal gas, and carbon monoxide at different temperatures, and the effects observed microscopically and by mechanical tests. The oxygen was always present as small globules of cuprous oxide uniformly distributed. The first observed effect of the action of reducing gases on copper containing oxygen was an apparent slight expansion of the envelopes of the oxide globules. This was followed by the formation of cracks which extended to form a network. In copper containing 0.07% of oxygen the effect of reducing gases began to be important at about 900° C. A markedly injurious effect was produced with even so low an oxygen content as 0.02%, but for a given effect a greater time and/or a higher temperature were needed than with higher percentages of oxygen. Coal gas was intermediate between hydrogen and carbon monoxide as regards depth of penetration in a given time, but the actual weakening effect seemed to be greater than with either of these other gases. Samples of copper containing small percentages of Zn, Mn, Al, or P, were entirely free from globules of cuprous oxide, and were not affected injuriously when heated in reducing atmospheres. Zinc would appear to be the most generally suitable deoxidiser, 0.10% being sufficient to confer complete immunity against the action of reducing gases, and for many purposes copper containing up to about 3% Zn is a better material than commercially pure copper.—T. St.

*Brass and other copper alloys; Season-cracking of —.*  
H. Moore, S. Beckinsale, and C. E. Mallinson. Inst. Metals, March, 1921. [Advance copy.] 91 pages.

THE season-cracking of brass appears to require both that the metal shall be in a state of stress and that it shall be subject to the action of some agent which selectively attacks the intercrystalline material. Normal corrosion, such as is produced by many acids, for example, does not give rise to season-cracking. The most usual agents are probably ammonia compounds derived from the atmosphere, but in studying the liability of brasses to develop cracks a 1% solution of mercurous nitrate is very convenient. A coating of lacquer is not a protection against season-cracking, but nickel-plating appears to give complete protection provided the coating is continuous and sufficiently thick. Surface defects do not appear to contribute to the development of season-cracks to any important extent. Season-cracks almost invariably follow an intercrystalline path, this being a most marked and characteristic feature. The greater the degree of hardening by cold-work, the less is the brass affected by the intercrystalline weaken-

ing action of ammonia or mercury. Liability to the development of season-cracks appears to diminish as the zinc content is reduced: copper and zinc-copper alloys containing less than about 10% Zn are unlikely to fail by the development of season-cracks in service. Within the limits of, say, 25 to 45% Zn little improvement in resistance to season-cracking is likely to be obtained by changes in composition or treatment, if the state of initial stress is not affected. The apparent advantage of harder worked brass is largely discounted by the higher stresses which are likely to exist in harder brass, if steps are not taken to remove them. A suitably controlled low-temperature annealing, which will remove stress without affecting the hardness, appears to be the most effective safeguard against failure by season-cracking, and might well be applied to all brass articles made by cold-work operations capable of inducing permanent internal stress. The authors consider that the results obtained by them afford strong support to the hypothesis of an intercrystalline material differing essentially in its properties from the crystals themselves, and that this material appears to be identical in its properties with Beilby's amorphous phase found in surface films. They have found no evidence which would indicate that season-cracking is the result of viscous flow of the intercrystalline cement. The undoubtedly intercrystalline character of season-cracks appears to be fully explained by the selective weakening action of a chemical substance on the intercrystalline material. In testing brass samples by the mercurous nitrate test for liability to develop season-cracks the standard method adopted by the authors was to pickle the specimen first for 30 secs. in a bath containing 40 vols. of nitric acid (sp. gr. 1.42) and 60 vols. of water, then to wash it thoroughly in water, and immerse it, without drying, in a solution made up with distilled water, containing 1 g.  $\text{Hg}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$  crystals and 1 c.c. of nitric acid (sp. gr. 1.42) in 100 c.c.—T. St.

*Copper with tin; Constitution of the alloys of —.*  
Parts III. and IV. J. L. Haughton. Inst. Metals, March, 1921. [Advance copy.] 22 pages. (Cf. J., 1915, 358.)

THE author has investigated the copper-tin alloys containing from 30 to 100% of Sn at temperatures below about 250° C. Thermal curves of the carefully annealed alloys were taken in a furnace arranged to give a uniform temperature gradient between 500° C. and 50° C., and in some cases electrical resistance measurements were also made. From the results the author has plotted an equilibrium diagram. In discussing the thermal curves obtained it is pointed out that the 210° C. and 190° C. arrests do not appear to be constant temperature arrests as is the case with the eutectic. The range of composition over which the 190° C. point is observed corresponds to the range in which the  $\epsilon$  constituent occurs, and the magnitude of the heat evolution increases with addition of tin, so far as can be observed, until the point at which the alloy consists of pure  $\epsilon$  (say about 60% Sn), after which it decreases in magnitude once more. This fact appears to point to a polymorphic change of  $\epsilon$  at 190° C., but no evidence as to the meaning either of the 210° C. arrest point or of that at 190° C. was obtainable from an examination of the microstructure of the alloys. Also, no evidence was obtained of the existence of the allotropic transformation in tin at 161° C. With regard to the extreme right-hand portion of the diagram, microscopical examination of alloys annealed at different temperatures leads the author to draw the limit of the solid solution of copper in tin at 99.8% Sn and 195° C., and to indicate a slight falling off of solubility with falling temperature. The



composition of the pure eutectic as determined by Heycock and Neville (J., 1896, 810) and recently confirmed by Guervich and Hromalko (Bull. Amer. Inst. Min. Met. Eng., 1919, No. 152), viz., 99% Sn, is accepted by the author, who, however, makes the melting point  $227.4^{\circ}\text{C.}$  as against  $227.1^{\circ}\text{C.}$  the value given by the latter investigators.—T. St.

*Copper alloys; Plastic deformation of some — at elevated temperatures.* C. A. Edwards and A. M. Herbert. Inst. Metals, 9.3.21. [Advance copy.] 25 pages.

THE indentations produced by a blow of 63 in.-lb. on test pieces of various copper-aluminium and copper-zinc alloys at temperatures between  $16^{\circ}$  and  $900^{\circ}\text{C.}$  were measured, and from these figures the Brinell hardness numbers were calculated. Graphs are given showing the relation between these numbers and the temperature for each of the alloys tested and the relation between the numbers and the composition of the alloys in each series. In general in both series the hardness diminishes with rise of temperature, the rate of falling-off depending on the composition of the alloys. In the zinc-copper series a sharp break in the curve is noticed at  $470^{\circ}\text{C.}$  in alloys containing more than 34% Zn, corresponding to a transformation in the  $\beta$  constituent. A similar break is noticed in the copper-aluminium series somewhere about  $550^{\circ}\text{C.}$  with alloys containing more than 12.4% Al.—A. R. P.

*Aluminium sheet; Stages in the recrystallisation of — on heating; with a note on the birth of crystals in strained metals and alloys.* H. C. H. Carpenter and C. F. Elam. Inst. Metals, 9.3.21. [Advance copy.] 22 pages.

THE structure of aluminium sheet (99.6% Al, 0.19% Si, 0.14% Fe) was examined during the various stages of rolling and after heating to temperatures from  $200^{\circ}$  to  $600^{\circ}\text{C.}$  for periods varying from 1 hr. to several months, and photomicrographs are given showing the changes that occur. On rolling, the original crystals of the cast metal are elongated in the direction of rolling but the crystalline structure of the metal does not disappear. Three distinct types of structure are produced on heating; the first visible change occurs when the metal is heated for long periods at  $200^{\circ}\text{C.}$  or for shorter periods at  $250^{\circ}$ – $300^{\circ}\text{C.}$  and is characterised by the general tarnishing of the surface, a granular structure, and the blurring of the original boundaries of the flattened crystals. If the metal is kept for a longer time between  $250^{\circ}\text{C.}$  and  $300^{\circ}\text{C.}$ , new crystals, which appear white, commence to form in the old crystal boundaries, and eventually the whole metal recrystallises, the crystals being very much elongated in the direction of rolling. If the heating is carried out at a higher temperature, the crystals, while still being slightly elongated, are approximately equiaxed. From an examination of the recrystallisation of a worked aluminium-zinc alloy and a sample of 70:30 brass it was concluded that all new crystals originate either in crystal boundaries or in twin boundaries and that they grow indiscriminately into the surrounding old crystals.—A. R. P.

*Spongy platinum; Preparation of —.* R. Feulgen. Ber., 1921, 54, 360–361.

A solution of chloroplatinic acid (5 g.) in water (5 c.c.) is mixed with formaldehyde solution (40%, 7 c.c.), and sodium hydroxide (5 g.) dissolved in water (10 c.c.) is added gradually. After remaining at the laboratory temperature for  $\frac{1}{2}$  hr., the mixture is heated for 15 mins. at  $55^{\circ}\text{C.}$  and then poured into a half-litre flask half filled with water. The latter is agitated violently for a few minutes which causes the precipitate to settle in coarse particles leaving an almost colourless supernatant

liquor. The latter is decanted and the precipitate is washed with water strongly acidified with acetic acid; coarse particles are again formed which can now be washed as is necessary without showing any tendency to pass into colloidal solution. The metal is finally collected and dried in a vacuum over sulphuric acid. Great caution must be observed in the subsequent admission of air into the desiccator as the metal readily becomes incandescent owing to absorption of oxygen. Previous to use it is advisable to grind and wash it once more. A very active catalyst is thus obtained.—H. W.

*Bearing metals.* B. Simmersbach. Chem.-Zeit., 1921, 45, 216–219.

IN an examination of the properties, and suitability for various applications, of gun-metal and white metal alloys, the influence of re-melting on their composition and characteristics has been investigated. With white metal, even after five fusions, no appreciable alteration in chemical composition was found; the copper was slightly higher, and the amounts of tin and antimony decreased, but the relative proportion of the last two metals remained unaltered. The temperature of fusion and the rate of cooling, however, appreciably affect the hardness and resistance of the alloy; the best results are secured by rapid cooling from a moderately high temperature. With gun-metal greater hardness was obtained by melting at  $900^{\circ}\text{C.}$  than at  $700^{\circ}\text{C.}$ ; no appreciable difference was detected for temperatures between  $400^{\circ}\text{C.}$  and  $700^{\circ}\text{C.}$  The critical temperature is  $782^{\circ}\text{C.}$ ; at or above this temperature rapidity of quenching the melt is of importance, and has a greater influence on the properties of the alloy than the amount of tin which it contains. Various applications of both gun-metal bronzes and white metal alloys are discussed, and many compositions for the latter are given.—W. J. W.

*Calcium; Some notes on —.* P. H. Brace. Inst. Metals, 9.3.21. [Advance copy.] 16 pages.

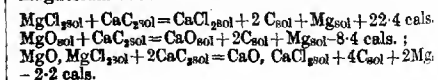
A HISTORICAL survey of the development of the metallurgy of calcium is given, together with the most important properties and applications of the metal. A method for the production of the metal from fused calcium chloride is described (cf. J. 1920, 370 A). Emphasis is laid on the necessity for using a very pure anhydrous chloride, which should not contain more than traces of sodium, magnesium or aluminium chlorides.—A. R. P.

*Gallium: its electrolytic behaviour, purification, melting point, density, coefficient of expansion, compressibility, surface tension, and latent heat of fusion.* T. W. Richards and S. Boyer. J. Amer. Chem. Soc., 1921, 43, 274–294.

THE single electrode potential of gallium is not easily reproducible, and has a maximum value of  $-0.30$  volt in  $N/10$  solution. Gallium is purified by electrolysis, followed by ignition in a high vacuum or by recrystallisation to remove traces of zinc. It melts at  $29.75^{\circ}\text{C.}$ , and the solid has sp. gr. 5.90; the liquid 6.095, both at the melting point. The compressibility of the solid is 0.0000020 and of the liquid about twice as large. The coefficient of cubical expansion of the solid is 0.000055, and the sp. gr. at  $20^{\circ}\text{C.}$ , 5.907. The latent heat of fusion is 19.04 cal. per g. at 173 megabars pressure. (C. J. C.S. April.)—J. F. S.

*Reactions producing magnesium.* C. Matignon. Comptes rend., 1921, 172, 381–383.

THREE possible processes for the preparation of magnesium are:—



These processes can be made to yield magnesium, but the second only proceeded very slowly at 1300° C. with a specimen of calcined dolomite. The first reaction requires the preliminary preparation of anhydrous magnesium chloride, which is a delicate and costly process. Attempts to apply these processes on a semi-industrial scale were not successful.

—W. G.

*Thermo-electric determination of transition points [of metals].* A. Smits and J. Spuyman. Proc. K. Akad. Wetensch., 1921, 23, 687—690.

WHEN the E.M.F. of a thermocouple is plotted against temperature, a smooth curve is generally obtained, but if one of the metals of the couple has a transition point within the temperature range considered, a discontinuity appears in the curve at the corresponding temperature. This phenomenon affords a rapid and exact method for determining transition temperatures. With an iron-tin thermocouple, the transition point of tin was indicated by a sharp break in the curve at 200.2° C. With a copper-tin thermocouple the transition temperature found was 200.5° C., but in this case the break was less sharp and the result possibly less accurate.—E. H. R.

*Thermo-elements. I. Thermal and electrical conductivities of copper-phosphorus alloys.* G. Pfeiderer. Ges. Abhandl. Kennt. Kohle, 1919, 4, 401—426. Chem. Zentr., 1921, 92, 1, 348—349.

THE efficiency of a thermo-element depends in an important degree on the ratio of the thermal to the electrical conductivity. The greater the thermal conductivity in proportion to the electrical, the lower the efficiency. According to the Wiedemann-Franz law, the ratio should be constant, but there are many known exceptions to this rule, and all of them are in the unfavourable direction. Experiments were made with copper-phosphorus alloys prepared from pure electrolytic copper, containing 0.68 to 1.98% P. No deviations from the above law in the favourable direction were found, and it is concluded that there is no hope of increasing the sensitiveness of thermo-elements by decreasing the thermal-electrical conductivity ratio.—E. H. R.

*Thermo-elements. II. Thermo-electric force of some antimony-cadmium alloys.* F. Fischer and G. Pfeiderer. Ges. Abhandl. Kennt. Kohle, 1919, 4, 440—447. Chem. Zentr., 1921, 92, 1, 349.

ALLOYS of antimony and cadmium in approximately equal atomic proportions show abnormally high thermo-electrical effects against metals or alloys. The effect has been found to be subject to great variations, and is extremely sensitive to heterogeneity in the composition of the alloy. The thermal treatment to which the alloy has been subjected is of great importance. At higher temperatures the thermo-electric force changes not merely as a function of temperature but also on account of permanent changes in the alloy. Amalgamation always lowers the thermal effect. Alloys which have been fused under a LiCl-KCl flux show a specially low effect, which can, however, be raised by subsequent warming without the salt layer.—E. H. R.

*Iodine and metals.* Matignon. See VII.

#### PATENTS.

*Steel; Production of rustless articles of* —. W. B. Ballantine. E.P. 158,329, 29.10.19.

NON-CORRODIBLE or rustless steel is cast around a hot core or ingot of ordinary steel. In the case of tubes rustless steel is cast on the outer and inner surfaces of a hollow cylindrical ingot.—T. H. Bu.

*Iron or steel; Pickling* —. British and Foreign Chemical Producers, Ltd. From Rheinische Kampferfabr. Ges. E.P. 158,768, 10.12.19.

TO avoid brittleness and the increased tendency of the pickled material to rust there is added to the pickling liquor a small proportion of crude anthracene, sulphite lye, or residues from the production of naphthalene, or a suitable extract thereof, an acid resin, waste acid from the refining of hydrocarbons, or a residue of the distillation of an organic compound.—T. H. Bu.

*Iron; Process for the anodic treatment of* —. E. Liebreich. G.P. 328,582, 31.1.19.

THE treatment is carried out at high temperatures, and solutions of chromium salts or chromic acid are employed as electrolytes. Bluish-white iron which has been rendered passive in cold nitric acid soon loses its passivity and regains the colour of ordinary iron. Iron rendered passive anodically at higher temperatures shows a white silvery colour which is stable in the air, provided the surface is not exposed to exceptional rust-producing influences. It may even be sand-papered and polished without losing its silvery colour.—J. H. L.

*Galvanised iron; Process for reducing the amount of zinc ashes and hard zinc produced in the manufacture of* —. C. Heitmann. G.P. 328,775, 24.10.19.

IRON articles, after removal from the pickling bath and before being galvanised, are subjected to compressed air in a fine stream or streams, whereby loosely adhering pieces of scale or iron are removed and are therefore prevented from rendering the zinc bath impure.—A. R. P.

*Selenium and noble metals from electrolytic slimes and the like; Process for recovering* —. M. Chikashige and D. Uno. E.P. 134,536, 28.10.19. Conv., 28.10.18.

THE electrolytic slime or other product is fused with lead and suitable alkalis in the form of hydroxide, carbonate, or nitrate, the noble metals being obtained as an alloy with lead and the selenium in combination with the alkali metals.—J. W. D.

*Ores and other materials; Leaching and other treatment of* —. E. T. Middlemiss. E.P. 158,320, 29.10 and 19.11.19.

FINELY crushed materials are treated with liquor in a series of open cylindrical vessels arranged in a circle, each vessel being inclined at an angle of 45° to the vertical. Each vessel is supported about its longitudinal axis and also by its lower edge when in the inclined position, so that it can be rotated about its longitudinal axis; and is also supported axially about a horizontal shaft so that it can be tilted. Means are also provided for moving the vessels bodily round the circle. By an arrangement of fixed cams the vessels, as they travel round the circle, are automatically raised from their normal angular positions to the vertical for charging, or lowered in the reverse direction for discharging. The crushed material in the vessels is bodily rotated and each particle on exceeding the angle of repose rolls down over the other particles in a spiral path while exposed to the action of the liquor which is more or less quiescent.—J. W. D.

*Silver-plating powder.* Y. A. F. Schwartz. E.P. 158,400, 6.2.20.

A PHOSPHATE of silver, preferably tribasic silver orthophosphate, is mixed with an organic acid soluble in water, preferably tartaric acid, and a chemically indifferent substance, such as kieselguhr.—T. H. Bu.

**Metal powders; Manufacture of** —. W. P. Heskett. E.P. 158,740, 21.11.19.

DIFFERENT metals (e.g., Mn 40%, Fe 10% Cu 10%, Al 10%, Sn or Bi 5%, Zn 15%, and Pb or Sb 10%) are subjected to heat so as to melt and alloy them without volatilisation and/or oxidation of any of the metals. The alloyed mass on cooling disintegrates into fine powder.—J. W. D.

**Alloy.** F. Milliken. Assr. to F. Milliken, S. F. Weaver, and J. M. Repplier. U.S.P. 1,350,166, 17.8.20. Appl., 29.8.18.

AN alloy characterised by a relatively high electrical resistance, elastic limit, tensile strength, and density, a close and fine grain, and a high resistance to corrosion, is composed of copper 50–60, nickel 26–34, iron 4–8, zinc 7–11%, and a purifying agent. The purifying agent preferred is commercial manganese-titanium (Mn 60, Ti 40%) and it is used in such proportion that 0.25–5% remains in the alloy.

**Contact points; [Alloy for] electric** —. J. H. Muller. U.S.P. 1,367,752, 8.2.21. Appl., 23.12.18.

CONTACT points for electrical purposes are made of an alloy containing Au 90%, Ni 10%.—C. A. K.

**Electrocyanide chlorination process; Machine for** —. R. T. Sill. U.S.P. 1,368,362, 15.2.21. Appl., 16.4.19.

A COMBINATION of a tank, two electrode plates within the tank, one above the other, a hollow column leading upwards from the central part of the space between the electrodes and discharging above the upper electrode, and means for raising liquid pulp through the column.—J. W. D.

**Precious metals; Precipitating and recovering** — from their solutions. K. B. Moor and H. R. Edmonds. U.S.P. 1,368,520, 15.2.21. Appl., 21.9.17.

THE process is continuous and consists in feeding finely ground charcoal through a series of extractor units, through which the metal-bearing solution passes in the opposite direction.—T. H. Bu.

**Metallurgical process. [Chloridising copper ore.]** R. H. Bradford. U.S.P. 1,368,885, 15.2.21. Appl., 18.11.18.

COPPER ore is mixed with a quantity of an alkaline-earth chloride containing sufficient chlorine to combine with the amount of copper present, and the mixture is heated in a non-reducing atmosphere to approximately 800° C. The copper is thus converted into cupric chloride which is volatilised, the fume being collected in the dry state, mixed with an alkaline-earth material and a carbonaceous reducing agent and heated to somewhat above the melting point of copper. Molten copper separates from the chloride slag, which may be re-used in the first part of the process.—T. H. Bu.

**Metallic values from ores; Process for obtaining** —. W. A. Schmidt, Assr. to International Precipitation Co. U.S.P. 1,368,973, 15.2.21. Appl., 2.2.20.

THE ore is heated and a metallic chloride is added at successive stages, the temperature being high enough to chloridise the metallic content of the ore and volatilise the resulting chloride.—T. H. Bu.

**Iron or steel; Alloys particularly for use in treating molten** —. E. C. R. Marks. From Union Carbide Co. E.P. 152,840, 11.9.19.

SEE U.S.P. 1,322,158 of 1919; J., 1920, 30 A.

**Steel; Manufacture of sound ingots and billets of special** —. R. A. Hadfield. U.S.P. 1,367,545, 8.2.21. Appl., 21.5.19.

SEE E.P. 131,373 of 1918; J., 1919, 826 A.

**Briquetting ores.** W. Tyrrell. E.P. 144,276, 31.5.20. Conv., 29.5.19.

SEE U.S.P. 1,356,100 of 1920; J., 1921, 16 A.

**Magnetic separator.** G. Ullrich, Assr. to The Chemical Foundation, Inc. U.S.P. 1,366,979, 1.2.21. Appl., 21.1.16.

SEE E.P. 100,062 of 1916; J., 1917, 600.

**Concentrating ore and the like; Method of and apparatus for** —. G. S. A. Appelqvist and E. O. E. Tydén. U.S.P. 1,367,223, 1.2.21. Appl., 31.12.13.

SEE E.P. 402 of 1913; J., 1913, 430.

**Copper sweeps and scraps; Process for the treatment of** —. C. C. Cito. U.S.P. 1,367,768, 8.2.21. Appl., 10.4.19.

SEE E.P. 123,418 of 1918; J., 1919, 261 A.

**Ore separating devices [; Flotation —].** A. H. Jones. E.P. 158,708, 11.11.19.

**Metallurgical filter.** U.S.P. 1,368,618. See 1.

**Gas for welding etc.** E.P. 158,633. See II A.

**Tunnel furnaces.** E.P. 157,474–5. See VIII.

**Electric furnaces.** E.P. 157,051. See XI.

## XI.—ELECTRO-CHEMISTRY.

**Ozonisers; Extrapolation and calculation of the concentration and yield of** —. H. Becker. Wiss. Veröffentlich. Siemens-Konzern, 1920, 1, 76–106. Chem. Zentr., 1921, 92, II., 253–254.

THE efficiency of an ozoniser is determined by the concentration and yield of ozone obtained when the apparatus is worked under definite conditions of frequency, tension, temperature, humidity of the air or oxygen employed, and velocity of gas in the ozoniser. In order to reduce the time necessary for the determination of the characteristic curves of an ozoniser, showing the relations of the concentration and yield of ozone respectively to the gas velocity, a method whereby the efficiency of the ozoniser at high velocities may be extrapolated from results obtained at low velocities is described. For certain velocities, the expenditure of energy in the ozoniser appears to be constant. A comparison of two ozonisers can be made on their respective expenditure of energy per unit volume of reaction space, which may be termed the wattage density. It is recommended that where alternating current of 500 frequency is available, or where the cost of electrical power is low, the ozoniser should be worked at the highest wattage density possible. Where alternating current of frequency 50 only is available, a small wattage density should be chosen for working the ozoniser. Experiments showed that from 30–35% of the energy supplied to an ozoniser was absorbed by the glass walls of the apparatus. This loss diminished with continued working of the ozoniser. When the wattage density employed exceeded a certain value, the concentration of ozone diminished.

—J. S. G. T.

**Nitrogen fixation.** Harding and McEachron. See VII.

## PATENTS.

*Crucible furnaces; Electric* —. C. Soncini. E.P. 142,836, 4.5.20. Conv., 25.4.19.

IN an electrically heated crucible furnace in which the resistance element is sectional and is composed of granular material, contact is made preferably by means of hollow water-cooled contact pieces, which, by means of a screw, may be caused to exert more or less pressure on the granular filling and so alter the resistance uniformly.—C. A. K.

*Electric furnaces.* Soc. Metallurgique du Frayol. E.P. 157,051, 22.3.20. Conv., 7.1.20.

IN a furnace for melting and refining metals and other substances, the arch, composed of graphite blocks, is capable of upward or downward movement in a water-cooled jacket of steel or cast iron, lining the upper part of the furnace. The electrodes are movable and lateral polar plates connected in parallel with a polar plate at the bottom of the furnace are provided in order to allow of easy starting. When charged cold, the furnace is inclined so that the charge is in contact with the lateral polar plates until the sole becomes heated and sufficiently conductive. On restoring the furnace to normal position, the lateral polar plates emerge from the bath.—C. A. K.

*Electric furnace for treating gases.* B. Thomas. U.S.P. 1,367,842, 8.2.21. Appl., 30.4.17.

THE arcing chamber of an electric furnace for treating gases is provided with a central air inlet at one end and an annular discharge opening at the other end. A number of electrodes bent into the form of a loop enter and leave the chamber through the air inlet. Another electrode is arranged within the annular exit.—J. S. G. T.

*Furnaces for the manufacture of electrodes and arc carbons; Collection and utilisation of tar vapours in —. Production of a protective coating for electrodes during the process of baking.* F. K. Meiser. G.P. (A) 329,185, 7.1.16, and (B) 329,539, 16.9.19.

(A) TAR vapours are exhausted from the furnace by injector action exerted by the flue gases, and are introduced into the producer used for supplying the gas for firing the furnace. (B) A flux, such as water-glass or common salt, is added to the protective layer (preferably only on the surface), so that it retains its porosity until the temperature at which tar vapours are evolved is attained, and then frits or melts to a dense surface layer.—J. S. G. T.

*Electrolytic gas generators [; Separator for —].* I. H. Levin. E.P. 158,148, 31.12.19.

A SEPARATOR for use more particularly in the manufacture of pure oxygen and hydrogen by electrolysis comprises a casing partly submerged in the electrolyte and in open communication at the top with the gas space above the electrolyte. A tortuous passage is formed by a partition dividing the casing into two branches communicating at one end. A tubular member open at both ends and extending at one end through the wall of the casing provides communication between the main body of liquid in the cell and the liquid within the casing. The electrolyte flows from one compartment of the cell through the tubular member and tortuous passage, and then either to a duct leading to a fill cup or other storage device, or through an opening in the diaphragm into the other compartment of the cell, and during its flow through the separator is completely freed from gas bubbles.—J. S. G. T.

*Electrolytic cell.* H. I. Allen, Assr. to Electron Chemical Co. U.S.P. (A) 1,368,010 and (B) 1,368,011, 8.2.21. Appl., 29.4.20.

(A) IN an electrolytic cell an anode consisting of parallel spaced bars is enclosed within a basket-shaped cathode formed with perforated corrugations which register with the bars. A porous diaphragm disposed between the anode and cathode bears against the inner faces of the corrugations. (B) A cathode for electrolytic cells consists of a metal plate having a flat marginal flange on all its sides, and provided with parallel corrugations closed at their ends by walls integral with the plate. The end walls and corrugations are perforated, while the flange is non-perforated.—J. S. G. T.

*Electric batteries; Primary* —. B. F. S. Baden-Powell. E.P. 158,733, 20.11.19.

IRON and carbon are employed as the respective electrodes in a primary battery in which water is the electrolyte. The carbon electrode may be surrounded by a depolariser, such as a mixture of carbon and manganese dioxide.—J. S. G. T.

*Galvanic cells; Exciting medium for use in —.* C. Beyer. G.P. 329,184, 23.4.20.

A MIXTURE of 90% of common salt, 5% of borax, and 5% of hexamethylenetetramine possesses the advantage over ammonium chloride that no crystals are deposited therefrom upon the zinc electrode.—J. S. G. T.

*Ozonising substances; Apparatus for —.* A. J. Moisant. U.S.P. 1,368,346, 15.2.21. Appl., 15.3.19.

AN apparatus for producing compounds containing ozone, comprises an ozone generator, means for supplying air under pressure thereto, a feeder for the substance to be ozonised, a mixing chamber, conduits connecting the parts and pressure-regulating valves disposed therein.—J. S. G. T.

*Ozone generator.* E. L. Joseph. U.S.P. 1,368,560, 15.2.21. Appl., 19.7.20.

SEE E.P. 128,867 of 1919; J., 1919, 644 A.

*Electrolysing salt solutions; Apparatus for —.* T. Matsushima. U.S.P. 1,368,955, 15.2.21. Appl., 18.3.18.

SEE E.P. 114,623 of 1918; J., 1919, 361 A.

SEE also pages (A) 205, *Electrical purification of gases* (G.P. 329,062). 216, *Nitrates* (U.S.P. 1,368,019). 223, *Electrolytic slimes* (E.P. 134,536). 224, *Electric contact points* (U.S.P. 1,367,752); *Electrocyanide chlorination process* (U.S.P. 1,368,362). 234, *Modifying starch* (U.S.P. 1,366,653).

## XII.—FATS; OILS; WAXES.

*Glycerides; Elucidation of the constitution of —.* A. Grün and F. Wittka. Ber., 1921, 54, 273—289.

ATTEMPTS to elucidate the constitution of mono- and di-glycerides by a study of the products of their oxidation are described.  $\alpha\beta$ -Diglycerides would be expected to pass through the aldehydes to the diacylglyceric acids, whilst  $\alpha\alpha'$ -diglycerides should yield derivatives of dihydroxyacetone. This is shown to occur, to a moderate extent at any rate, and, although it has not been possible to isolate the pure ketone in any instance, its presence can be detected and its amount estimated by Benedikt and Strache's method (treatment with an excess of phenylhydrazine and estimation of the amount of the latter unused by oxidation with Fehling's solution and measurement of the nitrogen evolved). The oxidation method is of no value for distinguish-

ing between  $\alpha$ - and  $\beta$ -monoglycerides, owing to the instability of the acyltartronic acids, which should be the primary oxidation products of the latter. The fat is suspended in glacial acetic acid and oxidised at 35° C. with finely divided potassium permanganate, which is added very gradually. A distearin, m.p. 74.5° C. after preservation, prepared from  $\alpha\alpha'$ -dichlorohydrin and potassium stearate, proved to be  $\alpha\alpha'$ -distearin mixed with a little of the  $\alpha\beta$ -variety. A second distearin, m.p. 77.5° C., prepared from  $\alpha$ -monochlorohydrin and stearyl chloride and subsequent treatment of the product with silver nitrite, was found to consist of almost equal proportions of the  $\alpha\alpha'$ - and  $\alpha\beta$ -isomerides. The progressive hydrolysis of tristearin by sulphuric acid gives a mixture of unchanged tri- and di- and monostearins. Oxidation of the distearins of higher and lower melting points shows the presence of the  $\alpha\alpha'$  variety in each. (Cf. J.C.S., April.)—H. W.

*Fats; Interchange of alkyl groups and its relationship to the constitution of —.* A. Grün, F. Wittka, and J. Scholze. Ber., 1921, 54, 290–299.

INTERCHANGE of alkyl groups between a fat and an alcohol can occur in the absence of catalyst at a sufficiently high temperature. Thus, when tristearin is heated with ethyl or isoamyl alcohol at 200° C. or 270° C. in a closed vessel, an equilibrium mixture is ultimately formed containing ethyl or isoamyl stearate, mono-, di-, and tri-stearins, and glycerol, part of the latter, however, suffering decomposition. Similarly, when a well-stirred mixture of ethyl or isoamyl stearate and glycerin is heated in an open vessel at 270°–280° C. (the liberated alcohol is volatilised during the process), stearin and alcohol are formed, and the reaction passes to completion. Even when an excess of glycerol is used, however, the reaction does not follow a simple course, since, owing to peculiar solubility relationships, distearin is alternately formed and decomposed. The anomalies observed in the melting-points of fats are explained on the assumption that they can exhibit co-ordination isomerism, existing as the simple ester form  $R.CO.OR'$  and the co-ordination form  $R.C \begin{smallmatrix} O \\ \diagup \end{smallmatrix} \dots R'$ . A monoglyceride can thus yield two isomerides, whilst a simple di- or tri-glyceride can exist in three or four forms respectively. This hypothesis is of wider applicability than that recently advanced by Fischer (Ber., 1920, 53, 1634) which is based on the possibility of slow interchange of alkyl groups at the ordinary temperature and in the absence of a catalyst. (Cf. J.C.S., April.)—H. W.

*Hydroxystearic acid; A new — from hardened castor oil.* H. Thoms and W. Deckert. Ber. deuts. Pharm. Ges., 1921, 31, 20–26.

By crystallisation of a "hardened" castor oil (m.p. 80° C., iodine value 12) from alcohol a glyceride was isolated which on saponification gave 1.12-hydroxystearic acid, m.p. 83° C. In the hydrogenation of castor oil, therefore, no displacement of the hydroxyl group of ricinoleic acid occurs. On dehydrating the new hydroxystearic acid with boiling 60% sulphuric acid, it yields a mixture of 11.12- and 12.13-iso-oleic acids.—G. F. M.

*Rubber seed oil, and a method of producing glycerides from fatty acids.* H. A. Gardner. Circ. 118, Paint Manufacturers' Assoc., U.S.A., Feb., 1921. 2 pp.

A SAMPLE of rubber-seed oil of iodine value (Hanns) 137 and acid value 57, when mixed with an amount of liquid cobalt drier sufficient to cause raw linseed oil to dry in 6 hrs., yielded a film which was not "set" but was still greasy after several days. On beating 50 g. of the oil with 2 g. of glycerin and 3 g. of calcium resinate to 260° C. for 90 mins., water

was given off, and the mixture darkened considerably. The resulting product of acid value 5.1, when mixed with the same quantity of cobalt drier as before, dried overnight to a film similar to that yielded by soya bean oil but inferior to that given by linseed oil. The method of esterifying with glycerin in presence of a catalyst, such as calcium resinate or tungate, may prove of value for improving highly acid oils.—A. de W.

*Tung oil heat test; Developments in the —.* H. A. Gardner. Circ. 119, Paint Manufacturers' Assoc., U.S.A., Feb., 1921, 8 pp.

THE finding of Jameson (J., 1920, 697 A) that the presence of free fatty acid has an important influence in retarding the gelatinisation of tung oil when examined by the Browne method (J., 1912, 731), as proved by the proportionately shorter time in which gelatinisation takes place after the free acids have been removed by calcium hydroxide, has been confirmed by the author. Jameson's method thus affords a means of determining the purity of samples of tung oil.—A. de W.

*Sulphonated oils; Determination of inorganic impurities in —.* R. Hart. J. Amer. Leather Chem. Assoc., 1920, 15, 404–406.

FROM the data obtained by the method described previously (J., 1917, 1139), together with determination of the ash, the amount of  $SO_2$  combined as sulphate and of other non-volatile impurities may be readily calculated.  $Na_2SO_4$  (due to combined  $SO_2$ ) =  $0.0634 (A_s + 2A)$ , where  $A_s$  represents the combined  $SO_2$  expressed in mg. of KOH, and  $A$  the alkali minus ammonia similarly expressed.  $A$  may be negative in the case of the presence of ammonium salts. The formula holds good only when  $2A < A_s$ ; when  $2A > A_s$ ,  $A_s$  is substituted for  $2A$ .  $Na_2CO_3$  =  $0.0473(2A - A_s)$ . Salt and impurities =  $Ash - Na_2SO_4$  (due to combined  $SO_2$ ) -  $Na_2CO_3$ . Calculated results agree very closely with actual determinations of Bumcke (cf. J., 1919, 831 A).—D. W.

*Sulphonated oils; Analysis of —. An improvement of the Hart method.* C. G. Bumcke. J. Amer. Leather Chem. Assoc., 1921, 16, 7. (Cf. J., 1917, 1139.)

10 c. of oil is boiled with 25 c.c. of  $N/1 H_2SO_4$  in a 500 c.c. Erlenmeyer flask fitted with a reflux condenser, for 1 hr. over a Bunsen burner. The use of  $N/1$  acid in place of  $N/2$  obviates the difficulties mentioned by Pickering (J., 1920, 305 r).—D. W.

*Soaps; Manufacture of — from paraffin wax and similar hydrocarbons.* W. Schrauth and P. Friesenhahn. Chem.-Zeit., 1921, 45, 177–178.

As substitutes for fatty acids, hydrocarbons such as paraffin have been successfully employed in the manufacture of soap. The constituents were beeswax, 350 kg., paraffin, 300 kg., and ceresin, 300 kg., to which 30 kg. of sodium hydroxide in 100 l. of water was added. The mixture was heated to 160°–180° C. in a steam-heated autoclave, at a pressure of 10 atm., with brisk mechanical agitation, for 8 hrs. A pressure of several atmospheres of air or oxygen is preferably maintained during the process, but the use of catalysts is unnecessary. The use of sodium carbonate, instead of hydroxide, is to be avoided as it gives rise to formation of anhydrides, lactones, or waxy substances, and consequent reduction of detergent properties.

—W. J. W.

*Emulsions. II. Reversal of phases by electrolytes, and effect of free fatty acids and alkalis on emulsion equilibrium.* S. S. Bhatnagar. Chem. Soc. Trans., 1921, 119, 61–68.

TRIVALENT electrolytes are more effective in bringing about the reversal of phases in emulsions than

divalent ones. The difference in the amount of electrolytes required to bring about the reversal with different soaps probably indicates a difference in their protective action, which decreases in the following order:—Potassium stearate, sodium stearate, sodium and potassium palmitates, potassium oleate, and sodium oleate. The reversal of phases in emulsions is affected by dilution in a similar way to the precipitation of colloidal sols. Attributing the cleansing power of soaps to their emulsifying efficiency, it will, on this view, be considerably affected by electrolytic impurities in the water, such as calcium salts. Traces of free fatty acid or of free alkali in emulsions shift the inversion point in opposite directions, and the oil globules are smaller in emulsions containing free alkali.—G. F. M.

## PATENTS.

*Fats and oils; Process of compounding* —. H. Wade. From Wilson and Co. E.P. 158,175, 17.5.20.

A "HARD" fat such as vegetable or animal stearine is melted with relatively large proportions of fluid oil (e.g. 1 pt. of stearine to 3–6 pts. of cottonseed oil, soya bean oil, etc.) and the mixture rapidly cooled during agitation to a temperature slightly above the m.pt. of the stearine present, e.g. 106°–115° F. (41°–46° C.). The mixture is then mechanically agitated while being cooled at a very slow rate to a temperature slightly below the temperature of crystallisation of the stearine, e.g. about 90° F. (32° C.) to produce a creamy homogeneous non-liquating mass which is then allowed to set. A small proportion of the solid stearine may be added as "starter" to the liquid mixture at or soon after the commencement of the slow cooling operation.—A. de W.

*Oil from coconuts; Process of extracting* —. *Process for the separation of food products from fresh coconuts*. W. Alexander, Assr. to The De Laval Separator Co. U.S.P. (a) 1,366,333 and (b) 1,366,339, 25.1.21. Appl., 17.10.18 and 21.5.19.

(A) The creamy emulsion separated from the liquid expressed from coconut pulp is inoculated with bacteria adapted to digest albumin, and the dry oil separated from the water and albumin sludge. (S) Coconut pulp is subjected to pressure, and the larger part of the water, together with a creamy emulsion consisting mainly of oil, is separated therefrom. The former forms an aqueous flavouring extract, whilst the emulsion is treated to eliminate the greater part of the water and contained albumin.—A. de W.

*[Fish] oil; Process for the extraction and refining of* —. P. M. Heyerdahl. U.S.P. 1,368,148, 8.2.21. Appl., 16.7.19.

The oil is treated below 100° C. with a stream of an inactive gas in the presence of sufficient water to wash the oil.—L. A. C.

*Fatty acids; Production of alkali salts of* — [from petroleum hydrocarbons etc.]. W. Schrauth. G.P. 327,048, 11.11.14.

SUBSTANCES obtained by the chlorination of petroleum fractions, preferably monochloro-derivatives, are subjected to fusion with alkali at 200°–300° C. in such a manner as to avoid a high pressure. The temperature is at first maintained at 200°–230°, and later raised to 300° C. The products obtained after 6–8 hrs. contain dry, odourless soaps, which are soluble in hot water and form an abundant lather. The excess of alkali may be neutralised with hydrochloric acid or fatty acids, or after re-melting it may be extracted by means of steam or water. The hydrogen evolved during the fusion

may be utilised technically. Cetyl chloride yields palmitic acid.—J. H. L.

*High melting-point fatty acids and neutral substances having an affinity for water; Process for the separation of* — from the saponification product of wool grease. I. Lifschütz. G.P. 329,232, 8.6.15.

THE product obtained by saponification of wool-grease according to G.P. 286,244 (J., 1915, 1153) is partly or completely washed and extracted with solvents for the unsaponifiable matter, the residual insoluble soap being treated with acids to separate the fatty acids. The yield of "hard fatty acids" and unsaponifiable matter amounts to about 50% of the wool-wax. The crystals which separate from the solution of the unsaponifiable matter form 15–20% of the total unsaponifiable matter, and consist of wool-grease alcohols of high melting point, which may be used alone or in conjunction with the bulk of the unsaponifiable matter as an artificial wax.—A. de W.

*Soap; Manufacture of* —. M. H. Ittner. U.S.P. 1,367,973, 8.2.21. Appl., 8.5.16.

STRONG solutions of soap in dilute lye are subjected when in a thin, freely flowing condition to a continuous centrifugal treatment whereby the separation of settled soap is effected.—A. de W.

*Detergent and fulling agent; Process for the preparation of a fat-free* —. E. Gips. G.P. 328,812, 15.9.16.

ANIMAL glue that has been hydrolysed by means of caustic alkalis is combined with fat solvents and saponins. Salts of laminaric acid, sodium or potassium carbonate, or ammonia may also be added.—A. de W.

*Glycerin; Process for decolorising liquids containing glycerin or crude* —. Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 144,727, 11.6.20. Conv., 12.2.19.

A CRUDE glycerin solution containing sulphuric acid is treated with oxalic acid, preferably in the form of powder, and then with barium carbonate. The addition of an amount of oxalic acid in excess of that required to precipitate the barium salt increases the bleaching action to such an extent as to avoid in some cases the necessity of adding absorbent decolorising material, e.g., animal charcoal.—A. de W.

*Oil; Processes of extracting* — from fatty substances. G. D. Rogers. E.P. 147,834, 9.7.20. Conv., 7.3.19.

SEE U.S.P. 1,326,968 of 1920; J., 1920, 240 A.

*Interaction of liquid or pulverulent materials*. G.P. 302,421. See I.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Pigments; Qualitative test for determining the texture of* —. H. A. Gardner and R. E. Coleman. Circ. 117, Paint Manufacturers' Assoc., U.S.A., Feb., 1921. 6 pp.

THE "texture" of a pigment relates to the structure of the particles, and is an important factor in regard to ease of grinding, character of finish, settling and hardening of the paint, and to some extent flowing and spreading power of the paint. To 1–2 g. of the pigment (depending on its oil absorption) placed on a ground-glass plate, is added 1 c.c. of a medium consisting of about equal parts of blown linseed oil (sp. gr. 0.945–0.950) and turpentine, adjusted to a viscosity slightly higher

than that of raw linseed oil. These are mixed to a thin semi-paste with a palette knife, followed by three grinding operations of 1 min. each with a 3" glass muller, in order to break down agglomerates and thoroughly wet each particle of the pigment. Between each mulling operation a few drops of the medium are added to compensate for evaporation or thickening. The resulting paste is thinned with the medium, transferred to a beaker, and further medium added until a total of 12 c.c. has been employed. After allowing to stand for 1–2 mins. to eliminate air, the mixture is poured on to the upper portion of a clean glass plate held in an almost vertical position in order that it may flow down, and then kept in a vertical position until the film is dry. An examination of the dried film shows the possible presence of larger particles, as well as pieces of lint and dirt in the lower portion of the film. The dispersive power of different liquids varies in degree, some liquids actually possessing the opposite effect of causing agglomeration of the pigment to large hard particles.

—A. de W.

*Nitrocellulose solutions for use in lacquers.* R. O. Herzog, A. Hildeshimer, and F. Medicus. Z. angew. Chem., 1921, 34, 57–60.

THE nature of the substances which are added to nitrocellulose solutions to produce pliability of the films, has considerable influence on the properties, such as elasticity, extensibility, tenacity, resistance to cold, and water absorption of the product. Investigations have been carried out with many of the agents used for this purpose, including esters of cinnamic acid and salicylic acid, ethyl phthalate, triphenyl and tricresyl phosphates, oils and fats, resorcinol acetate, sulphones, ether, ketones, form-anilide, and many combinations of these. By employing two or three of these agents together, certain characteristics may be obtained which are not secured by the use of one substance alone. Thus the brittleness of films, produced by the use of ethyl acetylsalicylate, may be avoided by also adding amyl cinnamate to the nitrocellulose solution. The figures for extensibility, with various additions, are given; they vary between 150% with amyl cinnamate and 25% with amyl salicylate. Ageing of films is sometimes due to solvent retained by the nitrocellulose; but chemical action between the nitrocellulose and the added substances, or changes in the latter, may be contributory causes.

—W. J. W.

#### PATENTS.

*Colours [; Lake —].* A. Linz, Assr. to Ultro Chemical Corp. U.S.P. 1,358,007, 9.11.20. Appl., 9.9.20.

AN alkali salt of the azo dyestuff from diazotised *p*-nitro-*p*-toluidine and 2-naphthol-3,6-disulphonic acid is dissolved or suspended in water and treated with barium chloride or other compound of a multivalent metal. The scarlet barium lake of the dyestuff is fast to light and insoluble in water, benzene, linseed oil, etc.

*Carbon black; Apparatus for the manufacture of —.* H. A. Bubb. U.S.P. 1,367,178, 1.2.21. Appl., 8.5.20.

A BURNER is arranged within and adjacent to one portion of a rotary barrel, and a hood fits in one end of the barrel. A scraper extending into the barrel removes the deposited carbon from the inner surfaces.—A. de W.

*Paint.* J. H. Gravell. U.S.P. 1,367,597, 8.2.21. Appl., 4.3.18.

AN anti-rusting paint for iron and steel contains calcium phosphates precipitated by alcohol.

—A. de W.

*Titanium oxide products [pigments]; Manufacture of —.* P. Farup, Assr. to Titan Co. A./S. U.S.P. 1,368,392, 15.2.21. Appl., 9.11.16.

A WHITE and relatively insoluble compound of a metal is precipitated upon particles of a titanium oxygen compound to stabilise it.—A. de W.

*Phenolic bodies; Condensation of — with aldehydic compounds.* Vickers, Ltd., Ioco Rubber and Waterproofing Co., Ltd., and W. H. Nuttall. E.P. 158,447, 15.12.19.

By applying hydrazine, instead of ammonia or the usual organic amino-compounds, as catalyst in the condensation of phenolic substances with aldehydic compounds, such as formaldehyde, the formation of highly coloured oxidation products is avoided and a colourless synthetic resin is obtainable.—D. F. T.

*Phenolic condensation products; Process of producing —.* I. V. Redman, A. J. Weith, and F. P. Brock, Assrs. to Redmanol Chemical Products Co. U.S.P. 1,368,753, 15.2.21. Appl., 6.6.18.

Loose fibrous material is impregnated with a phenolic substance and a methylene-amine substance which will react anhydrously therewith, and the mixture is subjected to forming and heat treatment until a hard, resistant, and substantially infusible material results.—A. de W.

*Plastic mass [from yeast].* W. Mooser-Schiess. U.S.P. 1,367,886, 8.2.21. Appl., 30.1.19. Renewed 27.12.20.

YEAST is mixed with esters of organic acids and polyhydric alcohols for the production of plastic masses.—D. F. T.

*Shellac and the like; Apparatus for recovering —.* T. W. Foote, Assr. to The Marble & Shattuck Chair Co. U.S.P. 1,368,426, 15.2.21. Appl., 2.6.17.

SHELLAC or the like is recovered from air charged with spray containing shellac by exhausting the air by means of a fan into a conduit provided with a baffle plate and screens at either end adapted to break up the spray, and thence into a collecting chamber.—A. de W.

*Phenol-formaldehyde condensation product and the production thereof.* A. W. Weller and W. T. Robinson-Bindley. U.S.P. 1,368,867, 15.2.21. Appl., 17.6.20.

SEE E.P. 152,384 of 1919; J., 1920, 792 A.

*Cellulose and artificial resin.* G.P. 328,783. See V.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Vulcanisation; Reactions of accelerators during —. II. Theory of accelerators based on the formation of polysulphides during vulcanisation.* W. Scott and C. W. Bedford. J. Ind. Eng. Chem., 1921, 13, 125–128. (Cf. J., 1920, 199 A.)

ORGANIC substances capable of accelerating vulcanisation may be classified into two groups, viz., hydrogen sulphide-polysulphide accelerators and carbosulphhydryl-polyulphide accelerators. The latter class comprises thioureas, dithiocarbamates, thiurams, mercaptans, disulphides, and certain Schiff's bases which during vulcanisation can give rise to thiourea derivatives. The former set includes all basic organic accelerators and such compounds as produce basic accelerators under the conditions of vulcanisation; in vulcanisation, hydrogen sulphide, arising from the action of sulphur on the rubber resins and proteins, combines with the basic accelerator with formation of a hydrosulphide which combines with free sulphur giving a polysulphide,  $RNH_2 \cdot SH_2$ ; the "polysulphide sul"



phur" is then transferred in an active form to the rubber. Hydroxides of the alkalis and alkaline earths also behave in this way; the theory explains their ineffectiveness with deresinised rubbers. Aldehyde-ammonia and *p*-phenylenediamine are purely hydrogen sulphide-polysulphide accelerators; hexamethylenetetramine, on account of the distinct behaviour of some of its decomposition products, can be placed in both groups. Litharge and zinc oxide are described as secondary accelerators, their function being merely to remove hydrogen sulphide from an organic hydrosulphide-polysulphide with consequent liberation of active sulphur and regeneration of the base which is then able once more to form polysulphide; secondary accelerators are not true catalysts.—D. F. T.

*Rubber; Action of certain organic accelerators in the vulcanisation of* —. III. G. D. Kratz, A. H. Flower, and B. J. Shapiro. *J. Ind. Eng. Chem.*, 1921, 13, 128—131. (Cf. *J.*, 1920, 378 A, 757 A.)

WITH a mixture of rubber and sulphur the effect of aniline is considerably greater than that of thiocarbonyl, whether comparison be made by means of the physical properties or of the coefficients of vulcanisation. In the presence of zinc oxide, however, both methods of comparison show thiocarbonyl to be more active than aniline. Although in the presence of zinc oxide, the tensile strength of the vulcanised product increases more rapidly with thiocarbonyl than with aniline, the maximum tensile strength is the same for each, and the vulcanisation coefficients corresponding with the maxima are practically identical. The maximum tensile strength for the rubber-sulphur mixture containing aniline is also the same as for the same mixture containing additional zinc oxide, but is attained at a much higher coefficient of vulcanisation. In the presence of zinc oxide, and of either accelerator, particularly of thiocarbonyl, the tensile strength develops in a remarkable manner during the earlier part of vulcanisation while the coefficient is still very low. The activity of aniline and thiocarbonyl appears to be of two distinct types; in the case of the latter a thiocarbamic acid is probably formed, capable of yielding a zinc salt of exceptional effectiveness. With aniline as accelerator, the effect of zinc oxide is manifested almost solely in the physical characteristics of the product, whereas zinc oxide greatly enhances the effect of thiocarbonyl from the point of view both of physical characteristics and chemical combination with sulphur.—D. F. T.

*Rubber energy*. W. B. Wiegand. *J. Ind. Eng. Chem.*, 1921, 13, 118—125. (Cf. *J.*, 1921, 123 A.)

WHEN rubber is stretched part of the energy is stored as potential energy, part is converted into "reversible" heat and part into frictional heat; on rapid retraction, the potential energy is almost entirely returned as work and the reversible heat is re-absorbed, whilst a further development of frictional heat occurs. The most important energy transformation is that of the conversion of work into frictional heat by hysteresis; the amount of this is found to be considerably increased on increasing the cyclic elongation and to be reduced by an increase in the degree of vulcanisation. Comparing various vulcanised rubber samples at a standard cyclic elongation, the hysteresis is found to be increased by the presence of fine mineral compounding ingredients, such as zinc oxide or carbon black, to a greater extent than by moderate changes in the degree of vulcanisation. Whereas a "pure" vulcanised rubber when led through a hysteresis loop to an elongation of 200% degrades approximately 4% of the total energy into heat, the heat formation is 8% in the presence of 5 vols. of zinc

oxide per 100 vols. of rubber, and 14% in the presence of 20 vols. of zinc oxide. In a pneumatic tyre cover the fabric contributes to the conversion of energy into frictional heat; the extent of the hysteresis loss from this cause may be determined by an electrical dynamometer or more simply estimated by inserting a 1-in. section of the carcass in the arm of a pendulum which is allowed to commence swinging from a definite position, the oscillation continuing longer the greater the resilience; in this way it is possible to examine the effect of alteration in the number of plies or type of fabric and in the quality and condition of the intervening layers of rubber. Other factors of importance affecting the energy phenomena of compounded rubber are the structure or the method of piling of the compounding ingredients and the surface tension between these and the surrounding rubber. (Cf. Schippel, *J.*, 1920, 199 A.)—D. F. T.

*Rubber seed oil*. Gardner. See XII.

#### PATENTS.

*Rubber; Process of devulcanising* —. J. Smith. E.P. 153,783 24.12.19.

THE rubber, in a fine state of division, is saturated with a cold solution of the hydroxide or carbonate of an alkali metal and is then heated in a closed receptacle until the internal pressure attains 100 lb. per sq. in. The mixture is then cooled and the soluble alkaline and saline matter removed by washing with water. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 11,159 of 1899, 17,313 of 1904, 2655 of 1905, 29,873 of 1912, and 28,167 of 1913; *J.*, 1899, 773; 1904, 1036; 1905, 934; 1913, 1078; 1914, 876.)—D. F. T.

*Rubber; Method of vulcanising* —. F. O. E. Stone. U.S.P. 1,368,071, 8.2.21. Appl., 30.4.20.

THE article to be vulcanised is immersed in a molten, easily fusible alloy; the alloy is then solidified by cooling and the embedded article vulcanised by submitting the solid mass to vulcanising conditions; after cooling the mass of metal is again fused by rapid heating, when the vulcanised article can be removed.—D. F. T.

*Vulcanised products; Production of soft and elastic* —. Farbenfabr. vorm. F. Bayer und Co. G.P. 323,610, 20.2.17.

PRODUCTS of great strength and extensibility are obtained by adding to the materials to be vulcanised metallic oxides or peroxides or organic or inorganic compounds which yield part of their oxygen at the temperature of vulcanisation, together with aliphatic or aromatic amines or ammonia compounds or their derivatives or salts. For example, antimony pentoxide, lead oxide, or lead peroxide may be added, together with diethylamine or aniline sulphate.—J. H. L.

*Lining tanks with hard rubber*. U.S.P. 1,367,231. See I.

#### XV.—LEATHER; BONE; HORN; GLUE.

*Chestnut wood; Extraction of* —. C. T. Gayley. *J. Amer. Leather Chem. Assoc.*, 1920, 15, 344—367.

EXPERIMENTS were made in a small copper autoclave heated by means of a steam jacket. The usual practice is to extract at a pressure of 2 atm. or more at 266° F. (130° C.). The yield of extract increases with increase of pressure up to 2 atm., but with higher pressures and at temperatures above 250° F. (about 120° C.) the tannin decomposes. The influence of temperature was determined by both the step-up and the constant temperature methods. In

the latter, after each addition of fresh water, the contents of the autoclave are heated to the same temperature, maintained at that temperature for a stated time, and the extract then drawn off. In the step-up method each decoction is carried on for 30 mins. and succeeding decoctions are carried out at temperatures rising 20° for each step. The step-up method of extraction yields a higher percentage of tannin by 5–15% for the same purity of extract. The author recommends this method with a range of 210°–280° F. (99°–138° C.) and 10° rise every time. There is a rapid decrease in the ratio of tans to non-tans extracted with increased time of decoction. The purity of the extract and the yield of tannin are almost uninfluenced by the amount of water used in each decoction. The extract of highest purity, the highest yield of extract, and the greatest speed of extraction are obtained from the finest shredded wood. Beyond a certain limit the increased yield obtained by a greater number of washings is more than counterbalanced by the diminished purity of the extract.—D. W.

*"Run" pelts; Investigation of the causes of — in the sweating process.* P. Hampshire. Bull. No. 2, Bureau of Bio-Technology, Leeds. J. Soc. Leather Trades' Chem., 1921, 5, 20–26.

The examination of "run" pelts damaged in the sweating process has shown the injury to be due to nematodes, organisms of a worm-like character living on organic fluids. The nematodes belong to the family *Nemathelminthes* and are found in all decaying vegetable and animal matter. They do not appear to be present in the skins on arrival at the tannery, but have been found in the sweating chamber itself. The infection arises from material in an advanced state of decomposition, and conditions should be aimed at in the sweating chamber to prevent putrefaction.—D. W.

*Acid unhairing [of hides].* R. H. Marriott. J. Soc. Leather Trades' Chem., 1921, 5, 2–9.

VARIOUS pieces of hide and skin were unhaird by means of dilute acids, with or without the addition of neutral salts, and the following conclusions are drawn from the results. The unhairing action is not bacterial and is not so effective as ordinary liming. The hair is not easily removed and the sweat and fat glands are still present in the treated material. Acids have a hating action and skin unhaird by means of acid is softer, when tanned, than a limed and bated skin. The acid liquor appeared to contain unhydrolysed gelatin and a ferment which produced alcohol. Acid unhairing is probably due to the hydrolytic action of the acid on some prosthetic protein similar to mucin, the carbohydrate group being attacked by the ferment.—D. W.

*Bating materials; Determination of the tryptic activity of —.* A. W. Thomas. J. Amer. Leather Chem. Assoc., 1920, 15, 221–228.

10 g. of casein is dissolved in 200 c.c. of distilled water and 8 c.c. of N/1 NaOH. The mixture is slowly heated to 40° C., and then rapidly to 85°–90° C. to destroy any proteolytic enzyme in the casein. The solution is cooled and made up to 500 c.c. 50 c.c. portions of this casein substrate are transferred each to a 100 c.c. flask, diluted to about 90 c.c., warmed to 40° C. in a thermostat, the enzyme preparation under examination added and the volume made up to 100 c.c. The enzyme action is stopped at stated intervals by pouring the mixture into 25 c.c. of 20% sodium sulphate solution and adding 5 c.c. of N/2 HCl. After standing for 1 hr. the undigested casein is filtered off and aliquot portions of the filtrate are used for measuring the proteolysis. One method consists in determining the nitrogen in a portion of the filtrate by the Kjeldahl

method, corrections being made for the nitrogen obtained by a blank determination and for the soluble nitrogen introduced with the enzyme preparation. The time of digestion should not be too prolonged since the product of weaker enzymes approaches that of the stronger at the end of 8 hrs. A more convenient and rapid method of measuring the proteolysis consists in titrating 50 c.c. of the filtrate with N/10 NaOH, using thymolphthalein as indicator. The amount of alkali required, less that used in a blank test, is an approximate measure of the proteolytic activity. Measurements should be made for the same sample with different times of digestion and compared with similar figures obtained with a standard material.—D. W.

*Hide; Swelling of limed and hydrated — in the presence of acid or alkali.* G. D. McLaughlin. J. Amer. Leather Chem. Assoc., 1920, 15, 228–234.

WEIGHED samples of plump, well hydrated, limed hide were soaked for 24 hrs. in equal volumes of solutions of various acids and alkalis of different strengths. With increasing concentration the swelling increased uniformly in the case of "weak" acids, whereas with strong acids it increased to a maximum, after which there was a rapid decrease. Further experiments showed there was no relationship between the concentration of the hydroxyl or hydrogen ions yielded by the dissociation of the various reagents in water and the amount of swelling of the hide. In explanation of the results it is maintained that protein lactate is produced from lactic acid formed in the green hide, sodium chloride proteinate is formed in the salting process and calcium proteinate in the liming process, and that each of these various protein compounds has a definite and characteristic hydration capacity. The increase in weight observed with increasing concentration of acid in the case of "weak" acids is explained by the absorption of the acid itself.—D. W.

*Plumping power of solutions [in tanning]; Measurement of the —.* A. A. Clafin. J. Amer. Leather Chem. Assoc., 1920, 15, 234–241.

THE volume of solution filtered from samples of hide powder shaken with different concentrations of acid is approximately constant for a given concentration. 200 c.c. of solution is shaken with 5 g. of hide powder, allowed to stand for 24 hrs., the liquor filtered through cotton cloth without squeezing, allowed to drain completely and the volume of the filtrate measured. The less filtrate obtained the greater the plumping power of the solution. The volumes obtained from 200 c.c. of different solutions are useful for comparative purposes. Lactic acid shows greater plumping power than acetic acid.—D. W.

*Sole leather; Laboratory wearing test to determine relative wear resistance of — at different depths throughout the thickness of a hide.* R. W. Hart. U.S. Bureau of Standards. Technol. Paper 166, 21.8.20. 7 pages.

TEST pieces from the same hide were divided into six groups, two of the original thickness of the leather, the others made thinner by skiving off respectively one-third from the grain surface, one-third from the flesh surface, two-thirds from the grain surface, and two-thirds from the flesh side. Preliminary experiments by subjecting these different pieces to the wearing test indicate that the outside surfaces of the leather have much less resistance to wear than the interior portions of the hide. The grain side of the leather has slightly more resistance than the flesh side. (Cf. report of British Boot and Shoe and Allied Trades Research Association).—D. W.

*Chromium in chrome [tanned] leather; Determination of —.* T. P. Hou. J. Amer. Leather Chem. Assoc., 1920, 15, 367—374.

In the sodium peroxide method of oxidising chromium solutions, mere boiling is not always sufficient to decompose the excess of sodium peroxide, especially when the volume of solution is large. It is recommended that the solution after oxidation with sodium peroxide should be gently evaporated to a small volume (10–15 c.c.), the evaporation repeated if necessary after addition of water, and the solution then diluted previous to titration. 5–10 c.c. excess of concentrated hydrochloric acid should be present when the solution is titrated. With these precautions the iodometric determination of chromium is rapid, the end point is very sharp, and the method is very accurate.

—D. W.

*Pyroxylin solvents and "leather solution": Valuation of —.* J. R. Lorenz. J. Amer. Leather Chem. Assoc., 1919, 14, 548—566.

The constituents of commercial "leather solutions" or "dopes" used in the production of pyroxylin-coated leather include amyl acetate, ethyl acetate, methyl ethyl ketone, "acetone oils," benzene, solvent naphtha, camphor oil, fusel oil, benzine, methyl and ethyl alcohols, soluble nitrocellulose, oxidised oils, pigments, and varnish gums. Methods for the determination of the various constituents of such mixtures are suggested.—D. W.

*Gelatin-hydrochloric acid equilibrium.* R. Wintgen and K. Krüger. Kolloid Zeits., 1921, 28, 81—89.

GELATIN and hydrochloric acid probably react according to the equation  $[\text{Ge}]\text{NH}_2 + \text{HCl} = [\text{Ge}]\text{NH}_3\text{Cl}$ , so that in aqueous solution the equilibrium  $[\text{Ge}]\text{NH}_2 + \text{H}_2\text{O} \rightleftharpoons [\text{Ge}]\text{NH}_3\text{OH} + \text{HCl}$  exists. The molecular weight of gelatin is 1014 and the hydrolysis constant  $4.139 \times 10^{-4}$ . (Cf. J.C.S., April.)—J. F. S.

*Sulphonated oils.* (1) Hart. (2) Bumcke. See XII.

*Vegetable glues.* Stern. See XVII.

#### PATENTS.

*Dressing hides; Method of —.* E. Kanet, Assr. to Tho Chemical Foundation, Inc. U.S.P. 1,367,054, 1.2.21. Appl., 28.6.16.

The hides are tanned by immersing them in a solution of ferric acetate in the presence of sodium chloride, at a low temperature, and heating the wet hide to cause separation of basic salts upon and between the hide fibres.—D. W.

*Leather or other goods; Apparatus for treating —.* W. J. Walker. U.S.P. 1,365,573, 11.1.21. Appl., 15.12.19.

See E.P. 134,040 of 1918; J., 1919, 955a.

*Dye substitute.* U.S.P. 1,367,862. See IV.

*Nitrogenous manures.* E.P. 147,798. See XVI.

### XVI.—SOILS; FERTILISERS.

*Soil moisture; New classification of the —.* G. Bouyoucos. Soil Sci., 1921, 11, 33—47.

The soil water is divided into gravitational, free, and unfree, the latter being further subdivided into capillary adsorbed, combined water of solid solution, and combined water of hydration. Of these forms, apart from the gravitational, the free water is that which freezes first at  $-1.5^\circ\text{C}$ ., the capillary-adsorbed water requires a temperature of  $-4^\circ\text{C}$ . before it freezes, whilst the combined

water does not freeze even at  $-78^\circ\text{C}$ . On the basis of this classification it was found that in some soils only one or two forms of water exist, while in others all three forms exist, but in different proportions. The dilatometer method (Michigan Agric. Exp. Stat., Tech. Bull. 36) is capable of measuring the relative amounts of these various forms of water in the soil. Repeated alternate freezing and thawing causes some of the unfree water to become free, the portion so changing belonging entirely to the capillary-adsorbed water. Of these various forms of water the gravitational and free water are freely available for the plant; the capillary-adsorbed water only slightly available, and the combined water not available.—W. G.

*Soil solution around the soil particles; Concentration of the —.* G. J. Bouyoucos. Soil Sci., 1921, 11, 131—138.

CONTRARY to the generally accepted view, the author believes that the solution around the soil particles and in the very fine capillary spaces is less concentrated than the mass of the solution. In support of this view he presents experimental evidence based on the diminution of the freezing-point depression of soils by successive freezing and thawing, the liberation of unfree (capillary adsorbed) water from soils by successive freezing and thawing, the abnormally greater increase in the freezing-point depression of soils as the moisture content decreases, and the equality in the freezing-point depression of the supernatant liquid and the soil which it bathes.—W. G.

*Soil reaction. I. A résumé. II. Colorimetric determination of the hydrogen ion concentration in soils and aqueous soil extracts.* E. A. Fisher. J. Agric. Sci., 1921, 11, 19—65.

A THEORETICAL paper in which the author criticises the various theories of soil acidity and the methods of determining the same, and in particular points out sources of error in the Hutchinson-McLennan method of determining the lime requirement of a soil (J., 1915, 565). Details of the application to soil extracts of the ordinary colorimetric method of determining hydrogen-ion concentration are given. For the determination of lime requirements of soils increasing amounts of barium hydroxide are added to the soil before making the extract, the hydrogen-ion concentration is determined in each case and a curve plotted. By interpolating at  $-\log [\text{H}^+] = 7.07$ , and converting barium hydroxide into lime, the lime requirement is obtained. The fineness of division of the soil is an important factor in these determinations of soil acidity.—W. G.

*Soils; Occurrence of different kinds of carbonates in certain —.* F. Hardy. J. Agric. Sci., 1921, 11, 1—18.

THE carbonate content of a soil is divided into "calcitoid" and "dolomitoid," the former being soluble in  $N/3$  acetic acid at the ordinary temperature and the latter insoluble. Marine silt soils may be expected to contain both forms of carbonate, but soils not directly or indirectly derived from marine silts usually do not contain dolomitoid carbonate. Cultivation for crop production reduces both the calcitoid and dolomitoid carbonate content of soils, but the former disappears much more rapidly than the latter. Soils containing both kinds of carbonate may show a "lime-requirement" as determined by the Hutchinson-McLennan method (J., 1915, 565), although the total carbonate content is apparently well above the usual value taken as indicating deficiency of carbonate. Pure dolomite appears appreciably to retard the whole process of nitrification, although magnesium carbonate only retards the second stage (nitrite to nitrate).—W. G.

*Soils; Chemical effect of salts on* — W. P. Kelley and A. B. Cummins. *Soil Sci.*, 1921, 172, 139—159.

WITH the two soils examined it was found that chemically equivalent solutions of the chloride, sulphate, and nitrate of a given base produce substantially equivalent chemical reactions. The solubility of the anion of the neutral salt solutions is not materially affected, but an exchange of bases takes place. The salts of the different bases may be arranged in an ascending order of reactivity—calcium, sodium, ammonium, potassium, magnesium. Calcium is the base most readily replaced from the two soils. When phosphates are used, considerable amounts of the phosphate-ion are precipitated by each of these soils. Chemical reactions take place between soils and alkaline solutions, whereby a normal carbonate is converted into a bicarbonate, the hydroxyl-ion concentration of the solution is lowered, and greater amounts of the cation of the solution are precipitated than is the case with neutral solutions. The reactions between neutral salts and soils are dependent on the concentration.—W. G.

*Humic acid; Preparation and fractionation of* — V. A. Beckley. *J. Agric. Sci.*, 1921, 11, 66—68.

THE soil or other starting material is extracted with 4% aqueous ammonia containing 2% ammonium chloride. The extract is filtered on a collodion filter, any organic matter not dissolving being classed as insoluble humin. The filtrate is acidified and humus is precipitated. Mulder's apocrenic acid remaining in solution. The humus is allowed to dry, and is then extracted with 95% alcohol, which dissolves out Hoppe-Seyler's hymatomelanic acid (*Z. physiol. Chem.*, 1889, 13, 62). The portion insoluble in alcohol is called humic acid, and is further fractionated by extraction with pyridine, a portion being soluble and the remainder insoluble in this solvent.—W. G.

*Humus; Formation of* — V. A. Beckley. *J. Agric. Sci.*, 1921, 11, 69—77.

THE formation of humus *in vitro* or in the soil proceeds in two stages. Carbohydrates react with mineral or amino-acids to give hydroxymethylfurfural, which then condenses to form humus. Under laboratory conditions furfural and levulinic acid are also formed. During the decomposition of cellulose by *Spirochaeta cytophaga* no hydroxymethylfurfural is apparently formed.—W. G.

*Sulphur and sulphur composts in relation to plant nutrition*. W. E. Tottingham and E. B. Hart. *Soil Sci.*, 1921, 11, 49—73.

EXPERIMENTS with soil composts having added sulphur or sulphur plus rock phosphate and similar composts with horse manure indicate that sulphur probably functions as a fertiliser both by oxidation to nutrient sulphate and by producing, through oxidation, an acid condition favourable to the production of available phosphate. These processes occur in composts of sulphur and rock phosphate, and probably also continue when the compost materials are incorporated into the soil. The use of sulphur as a fertiliser tends to deplete the soil of lime and phosphoric acid.—W. G.

*Sulphur; Inoculated — as a plant-food solvent*. J. G. Lipman, A. W. Blair, W. H. Martin, and C. S. Beckwith. *Soil Sci.*, 1921, 11, 87—92.

EXPERIMENTS with barley and potatoes as crops indicate that sulphur inoculated with micro-organisms capable of oxidising it (*cf. J.*, 1916, 1268; 1917, 227) is more effective than uninoculated sulphur for rendering inert mineral plant-food, such as the potash in greensand marl or the phosphate

in rock phosphate, accessible to growing crops. Mixtures of inoculated sulphur and ground rock phosphate gave better returns than rock phosphate alone.—W. G.

*Iron; Influence of — in the form of ferric phosphate and ferrous sulphate upon the growth of wheat in a nutrient solution*. L. H. Jones and J. W. Shive. *Soil Sci.*, 1921, 11, 93—99.

THE results of a limited number of experiments indicate that iron in the form of ferric phosphate is utilised very slowly and with difficulty by wheat plants. Ferrous sulphate, however, is readily available, but there is an optimum amount above which it is somewhat toxic.—W. G.

*Phosphate in soils; Short test for easily soluble* — O. M. Shedd. *Soil Sci.*, 1921, 11, 111—122.

THE air-dried soil (10 g.) is shaken for 5 mins. with 25 c.c. of N/5 nitric acid, the solution filtered, and the filtrate collected in a standard size test-tube. 1–2 c.c. of a 60% solution of ammonium nitrate and 5 c.c. of ammonium molybdate solution are added and the mixture is heated to 60°C., shaken several times, and allowed to stand for 30 mins. The volume of the precipitate is judged by the eye and classed as "large," "fair," "moderate," "very moderate," etc. The classification with different soils in this manner is in good agreement with the quantitative results obtained after the digestion of the soil with ten times its weight of N/5 nitric acid for 5 hrs. Any soil classed as very moderate to moderate or under, that is, containing 0.005–0.0075% or less of soluble phosphorus will probably respond to applications of phosphatic fertilisers.—W. G.

*Superphosphate; Determination of citrate-soluble phosphate in* — P. Müller. *Chem.-Zeit.*, 1921, 45, 178.

IN the estimation of citrate-soluble phosphate as magnesium ammonium phosphate the results are too low if the precipitate and solution are shaken for  $\frac{1}{2}$  hr. and then immediately filtered. It is advisable to let the precipitate stand overnight. In seven tests an average difference of 0.44% was obtained by the two methods.—W. J. W.

*Vitamins and auximones; Influence of — on vegetable growth*. A. Lumière. *Ann. Inst. Past.*, 1921, 35, 102—123.

VITAMINS are not necessary for plant growth, for this will take place in culture solutions of known chemical composition free from vitamins. Extracts containing the vitamins in a more or less altered condition may be beneficial to plant growth, but this action cannot be ascribed to the vitamins.

—J. C. D.

#### PATENTS.

*Fertilisers; Manufacture of artificial* — Akt.-Ges. für Anilin-Fabr. E.P. (A) 146,259, 28.6.20, and (B) 145,582, 29.6.20. Conv., 29.1 and 16.3.18.

(A) A CONCENTRATED solution (85%) of ammonium nitrate, at a temperature not below 70° C., is mixed with potassium chloride, or other inorganic salt with fertilising properties, previously heated to a high temperature, e.g., about 300° C. When reaction has occurred, and the product has hardened through evaporation of water, it is milled; it contains only 1–2% of water. (B) Instead of a hot inorganic salt, a cold salt may be employed, the heat of crystallisation alone sufficing to evaporate the water. Thus potassium sulphate, or a mixture of rock salt and calcined magnesium sulphate, may be added to the ammonium nitrate solution, the latter being at a temperature of 120° C.—W. J. W.

*Nitrogenous manures; Manufacture of — from waste leather and other animal detritus.* H. Devos. E.P. 147,798, 9.7.20. Conv., 17.12.13.

Waste leather or similar animal detritus is heated for 1–4 hrs. in a boiler with water containing a small amount of acid, without allowing the liquor to boil. This operation ensures deposition of a gelatinous mass at the bottom of the boiler without decomposition of its nitrogenous constituents. After decantation of the supernatant liquor, the material is allowed to solidify on slabs, and then placed on covered screens where hardening is completed, after which it is pulverised.—W. J. W.

*Fertiliser disintegrating and drying apparatus.* A. J. Case. E.P. 157,495, 16.8.19.

FERTILISING material mixed with straw and other foreign matter is introduced into an inclined drum formed of perforated plates and capable of being rotated, the interior of the drum having a series of inwardly curving blades. By their action straw and foreign matter are separated from the fertiliser and carried forward to the delivery end of the apparatus, whence they are removed by a conveyor. Finely-divided fertilising material which passes through the holes in the drum falls on to another conveyor. Coarser material is discharged into a trough from which it is elevated to a hopper above a disintegrator, and after passing through the latter it drops on to a table and is mechanically returned to the drum. Drying of the fertiliser is effected by means of a central steam pipe in the drum and a steam-heated plate underneath the disintegrator table.—W. J. W.

*Alunite ore; Utilisation of —.* A. Matheson. E.P. 158,293, 28.8.19.

ALUNITE ore containing 4–10%  $K_2O$  and 18–33%  $SO_3$  is broken or ground, and heated in a furnace having a series of hearths on which it is mechanically rahlled without admission of air. The sulphurous gases evolved are passed into a retort tower charged with calcined alunite and phosphate rock or bones. Interaction is completed by storing the treated material in open dumps, a potash phosphate fertiliser of any desired composition being produced.—C. I.

*Fertiliser; Production of a phosphate — containing potassium.* A. L. Kreiss. U.S.P. 1,363,580, 25.1.21. Appl., 30.7.20.

PHOSPHATE rock is mixed with a rock containing potassium and with more than one alkali metal salt, and the mixture is calcined and leached.—W. J. W.

*Fertiliser and process of producing the same.* F. S. Washburn, Assr. to American Cyanamid Co. U.S.P. 1,367,846, 8.2.21. Appl., 12.3.20.

PHOSPHATE rock is treated with not more than 10% of sulphuric acid; the phosphoric acid produced is separated from insoluble matter, mixed with sulphuric acid, and the mixture is neutralised with ammonia.—W. J. W.

*Fertiliser composition.* P. U. Ducommun. U.S.P. 1,368,249, 15.2.21. Appl., 14.7.19.

A BACTERIAL culture is prepared from farmyard manure combined with foods, including pulverised corn (maize) cobs, to retain moisture and sustain bacterial action.—W. J. W.

*Ammonium bicarbonate for fertilising purposes; Process for increasing the stability of —.* Badische Anilin u. Soda Fabrik. G.P. (a) 310,055, 11.6.18, and (b) 310,056, 30.6.18.

To prevent or retard separation of ammonia from

ammonium bicarbonate it is mixed with (a) 5–10% of anhydrous sodium sulphate or (b) 10% of magnesium sulphate (kieserite).—W. J. W.

*Boiling and drying organic substances, particularly offal; Apparatus for —.* K. Niessen. E.P. 137,828, 12.1.20. Conv., 11.5.15.

SEE G.P. 318,542 of 1915; J., 1920, 498 A.

*Potassium nitrate etc.* G.P. 310,601. See VII.

*Fish products.* E.P. 157,515. See XIX A.

*Destroying pests.* G.P. 329,201. See XIX B.

## XVII.—SUGARS; STARCHES; GUMS.

*Sugar; Loss of — occurring when sweet-water is used for the slaking of caustic lime.* P. Beyersdorfer. Z. Ver. deuts. Zucker-Ind., 1921, 75–87.

LABORATORY experiments lead to the conclusion that when the sweet-water from the washing of the filter-press cake is used for slaking the lime used in clarification, about 5 to 15% of the sugar present in it is destroyed, the actual amount depending principally upon the degree of heat resulting from the hydration, that is, upon the quality of the lime and the proportion in which it is added to the liquid. In practice, where little care may be taken to control the temperature resulting on slaking, the loss may be greater, probably averaging 20%. Caramel does not appear to be formed in any appreciable amount, lactic and acetic acids being the principal products of the decomposition, though benzaldehyde may result if the temperature be unusually high.—J. P. O.

*Beet sugar factory; Recovery of the ammonia evolved during clarification in the —.* J. Silhavý. Z. Zuckerind. Czecho-Slov., 1921, 45, 155–156.

In the operations of liming with 2% of  $CaO$  at  $80^{\circ}$ – $90^{\circ}$  C., and carbonating during 6–7 min., only 4.38 kg. of  $NH_3$  or 16.9 kg. of  $(NH_4)_2SO_4$  can be recovered in the case of a factory slicing 600 tons of beet per day. Donath's experiments (J., 1920, 379 A) leading to the expectation of a higher yield were carried out under conditions which did not sufficiently correspond to those obtaining in actual practice.—J. P. O.

*Sucrose, glucose [dextrose], and fructose [levulose]; Optical rotation of mixtures of —.* W. C. Vosburgh. J. Amer. Chem. Soc., 1921, 43, 219–232.

THE specific rotations of mixtures of dextrose and levulose in equal proportions (invert sugar) in solution are those which the sugars would have if each were present alone at a concentration equal to the total invert sugar concentration. The specific rotations of dextrose and sucrose in mixtures of the two are those which the sugars would have if each were present alone at a concentration equal to the total sugar concentration. The presence of a constant amount of hydrochloric acid of concentration  $N/10$ , sodium chloride  $N/10$ , or sodium carbonate  $N/25$  has no effect on the determination of the percentage of sucrose when the rotations of the pure sucrose and invert sugar are determined under the same conditions as in the case of the mixtures. (Cf. J.C.S., April.)—J. F. S.

*Cuprous oxide by reduction [with sugars].* V. V. Sarma. Chem. News, 1921, 122, 99–100.

THE yellow substance produced by the reduction of an alkaline cupric salt with dextrose is a mixture of cuprous oxide, cuprous hydroxide, and 2–3% of water. A small portion is soluble in ether.

Reduction with sucrose produces only red cuprous hydroxide. (*Cf.* J.C.S., April.)—J. F. S.

*Cellobiose [cellose]. Constitution of the disaccharides.* V. W. N. Haworth and E. L. Hirst. *Chem. Soc. Trans.*, 1921, 119, 193—201.

THE complete methylation of cellobiose, first with methyl sulphate and sodium hydroxide and finally with Purdie's reagents (methyl iodide and silver oxide), gave a crystalline heptamethyl-methylcellobioside, m.p.  $76^{\circ}$ — $78^{\circ}$  C.,  $n_D = 1.4643$ , which on hydrolysis with 5% hydrochloric acid at  $80^{\circ}$  C. yielded two products, one, soluble in light petroleum, and identified as tetramethylglucose of the butylene oxide type, and the other, soluble in ether, which was shown to be a trimethylglucose identical with that obtained by the hydrolysis of methylated lactose, and from methylated cellulose. That this trimethylglucose also possesses the butylene oxide structure was shown by conversion into the above tetramethylglucose by complete methylation to tetramethyl- $\beta$ -methylglucoside, followed by hydrolysis, and since the free hydroxyl group is the one adjacent to the terminal group, the provisional formula put forward for the constitution of cellobiose (J., 1919, 691A) in which the two glucose residues are linked together through the reducing group of the one and the hydroxyl group next to the  $\text{CH}_2\text{OH}$  group of the other, is confirmed.

—G. F. M.

*Glues; Vegetable — for wood.* E. Stern. *J. prakt. Chem.*, 1920, 101, 308—327.

THE products of the action of alkali on starch are efficient adhesives, but they gradually suffer coagulation, and it is doubtful whether the difficulty is entirely overcome by preliminary treatment of the starch with acid. Starch viscose (Cross and others, J., 1907, 628) does not coagulate, and experience has shown it to be an efficient substitute for ordinary joiners' glue and to remain unchanged for several months. Joints between wood made by mixtures of the viscose with 0.5, and 10% respectively of water hardened in 48 hrs. at  $13^{\circ}$ — $18^{\circ}$  C., and withstood strains of 47.7, 49.2, and 51.1 kg. per sq. cm., whilst a similar joint made with a mixture of bone glue with 1.5 pts. of water withstood 48 kg. per sq. cm. The paper is largely devoted to a discussion of the chemical nature of starch viscose and of the product of the action of alkali on starch. (*Cf.* J.C.S., April.)—J. K.

*Lactose.* Adriano. See XIXA.

#### PATENTS.

[Sugar] juice; Apparatus for the introduction of — into vacuum boilers and crystallising vessels. A. Grantzdörffer. G.P. 327,397, 20.8.19.

THE apparatus consists of a substantially rectangular box which has in its long side an outlet slot approximately parallel to the wall of the vessel, so that the juice entering the vessel through the slot causes a circulatory motion of the contents of the vessel.—J. H. L.

*Milk-sugar; Manufacture of —.* R. W. Mumford, Assr. to Refining Products Corp. U.S.P. 1,366,822, 25.1.21. Appl., 2.7.17.

IN the manufacture of milk-sugar from clarified and purified whey by evaporation and crystallisation, the liquid is treated with a vegetable carbon which retains the cellular texture of the original material (*cf.* U.S.P. 1,286,187, 1,287,592, and 1,314,204; J., 1919, 129A, 785A).—J. H. L.

*Starch; Process of modifying —.* E. H. Harvey, Assr. to Perkins Glue Co. U.S.P. 1,366,653, 25.1.21. Appl., 13.5.20.

AN electric current is passed through a conducting

bath containing the starch, and the treatment is arrested before the starch has been modified beyond the stage of soluble starch.—J. H. L.

*Filters.* E.P. 158,387. See I.

#### XVIII.—FERMENTATION INDUSTRIES.

*Yeast crops and the factors which determine them.* A. Slator. *Cbem. Soc. Trans.*, 1921, 119, 115—131.

IF conditions be so arranged that the limiting factor for the growth of yeast is the amount of sugar in the medium, then the yeast crop will be determined by the way the ratio  $\kappa/\epsilon$  ( $\kappa$  is the constant of growth and  $\epsilon$  the fermentative activity) varies with different concentrations of sugar, and a diagram can be constructed to show the crops with various initial concentrations of sugar. The method is of general application, and similar diagrams can be constructed when other limiting factors are predominant. The favourable influence of atmospheric oxygen on the crop is to be attributed more to the displacement of carbon dioxide than to a positively favourable influence of oxygen; in fact, free oxygen rather retards the growth in malt wort during the sugar fermentation. The retarding influence of carbon dioxide is much greater, however, and the aeration of the wort lessens the supersaturation by carbon dioxide and larger crops result. There is, however, a second mode of growth such as occurs in lactose yeast water, which is unaccompanied by alcoholic fermentation, and here free oxygen is essential; in the absence of oxygen hardly any growth occurs. The factors determining which mode of growth takes place when both are possible are not clearly defined, but the available nitrogenous food is probably of importance. The influence of temperature on yeast crops is small, the temperature coefficients of  $\kappa$  and  $\epsilon$  being not greatly different from each other. No evidence was forthcoming of the necessity of a vitamin to yeast growth.—G. F. M.

*Yeast; Proteins of —.* P. Thomas. *Ann. Inst. Past.*, 1921, 35, 43—95.

TWO proteins have been isolated from yeast. One appears to be a phosphoprotein resembling in some ways caseinogen, and this is termed zymocasein; the other is an albumin, cerevisine. Both proteins have been examined and their products of hydrolysis estimated. Particularly notable is the high percentage of tryptophane present. (*Cf.* J.C.S., April.)—J. C. D.

*Pseudomonas polysaccharidarum (n. sp.): an organism which decomposes complex polysaccharides.* A. Epstein. *Bull. Soc. Bot. Genève*, 1920, II, 11, 191—198. *Chem. Zentr.*, 1921, 92, I, 224.

THE new species readily decomposes pectin substances, cellulose, and starch, in presence of air, but does not attack lignin substances. In presence of air it liquefies gelatin within 5 weeks; the resulting liquid is acid and contains amines but not indole.—J. H. L.

*Emulsin; Preparation of — from almonds by different precipitating agents.* M. Bridel and R. Arnold. *Bull. Soc. Chim. Biol.*, 1920, 2, 216—222.

THE properties of the emulsin do not appear to be different when different precipitating agents, such as methyl or ethyl alcohol or acetone, are used, unless contact with the precipitating agent is prolonged.—J. C. D.

*Salicinase; Influence of heat on the activity of —.*  
G. Bertrand and A. Compton. *Comptes rend.*,  
1921, 172, 548–551.

THE values of the optimum and maximum temperatures of activity of salicinase vary inversely with the duration of the experiment, but the value of the optimum temperature reaches a minimum of 30° C. for a period of 32 hrs. and prolonging the experiment does not cause any further diminution. The temperature of maximum activity is 70° C. and corresponds with the temperature of instantaneous destruction of the enzyme by heat.

—W. G.

#### PATENTS.

*Lactic acid fermentation; Production of a raw material suitable for — from putrefied potatoes.*  
Knab und Lindenhayn. G.P. 326,548, 16.8.19.

COLLOIDAL constituents of the potatoes, which inhibit fermentation and are difficult to filter, are rendered innocuous by heating, the temperature employed being such that the starch is not gelatinised, e.g., 50°–60° C.—J. H. L.

*Yeast; Process for the regeneration of degenerated brewery —.* Nathan-Institut A.-G. G.P. 327,498, 14.9.16.

THE yeast is treated with a very dilute solution of alkali or alkaline earth, preferably a 0.05–0.25% solution, at 4°–7° C., and allowed to subside. The testinic acid or testilupin surrounding the cells is thus dissolved without injury to the cells. The yeast may afterwards be strongly aerated for 1 hr. in very dilute wort.—J. H. L.

*Alcohol and yeast; Production of — from sulphite-cellulose waste liquors.* F. Mizgajski. G.P. 329,111, 24.7.17.

THE waste liquors are mixed with molasses and fermented without addition of special yeast food.

—J. H. L.

*Plastic mass [from yeast].* U.S.P. 1,367,886. See XIII.

*Worts from vegetables etc.* G.P. 326,178. See XIXA.

*Foodstuffs from yeast.* G.P. 323,260. See XIXA.

### XIXA.—FOODS.

*Lactose [in milk]; Volumetric determination of by alkaline potassium permanganate.* F. T. Adriano. *Philippine J. Sci.*, 1920, 17, 213–220.

TWENTY-FIVE c.c. of N/10 permanganate solution, 25 c.c. of 0.848% sodium carbonate solution, 15 c.c. of water, and 10 c.c. of the lactose solution containing not more than 1.5 g. of the sugar (e.g., milk after treatment with copper sulphate, neutralisation, and filtration) are mixed, heated at 95° C. for 2 mins., 25 c.c. of 30% sulphuric acid is then added, and N/10 oxalic acid is run in until the liquid is clear. The excess of oxalic acid is titrated with N/10 permanganate solution. Reference to a table gives the amount of lactose corresponding with the quantity of permanganate used for the oxidation, this table having been prepared from results obtained with pure lactose. The results obtained are concordant but slightly lower than those yielded by the Soxhlet method.—W. P. S.

*Swelling ("solvation") in colloids [gluten, gliadin, barley meal]; Measurement of —.* H. Lüers and M. Schneider. *Kolloid. Zeits.*, 1921, 28, 1–4.

THE swelling of gluten and of its chief constituent, gliadin, in lactic acid solutions has been studied respectively by the gravimetric method by Upson

and Calvin (J., 1915, 629) and by the viscosimetric method by Lüers (J., 1920, 449 A). Comparison of the data obtained shows that the two methods give concordant results. The authors have now determined the swelling of barley meal in different aqueous media by the viscosimetric method and by the volumetric method (measurement of the height of a column of the meal after sedimentation in different media), and show that these methods also give concordant results. (Cf. J.C.S., March.)

—J. F. S.

*Hydrocyanic acid in Phaseolus lunatus beans; Quantity of —.* Lübrig. *Pharm. Zentralh.*, 1921, 62, 95–97. (Cf. J., 1920, 277 A, 382 A.)

SAMPLES from individual sacks of a truck load (200 sacks) of these beans yielded quantities of hydrocyanic acid varying from 2.3 to 37.7 mg. per 100 g. of beans; average, 20.1 mg. A composite sample from the whole consignment gave 26 mg. per 100 g., hence there is some difficulty in arriving at the true amount present. Beans containing relatively large quantities of the acid may be used for food provided that they are steeped in water for 25 hrs., and that the water in which they are cooked is rejected. There is no evidence that acid ferments (sour milk) produce further quantities of hydrocyanic acid from beans which have been freed from the acid.—W. P. S.

*Vitamin B; Effect of alkali on efficiency of —.*  
T. B. Osborne and C. S. Leavenworth. *J. Biol. Chem.*, 1921, 45, 423–426.

THE instability of this vitamin to alkalis reported by several previous investigators is confirmed.

—J. C. D.

*Yeast proteins.* Thomas. See XVIII.

#### PATENTS.

*Coffee; Manufacture of —.* E. C. R. Marks. From Floyd W. Robison Co. E.P. 157,493, 12.8.19.

UNROASTED coffee beans, softened by a limited treatment with water or steam, are subjected to the action of micro-organisms, preferably moulds, for several days, and the action is then arrested by roasting or otherwise. Selected organisms, e.g., *Aspergillus ochraceous*, or others from choice coffee, may be employed, and the beans may be first sterilised to exclude undesired types.—J. H. L.

*Fish products and the production thereof.* P. B. Jagger. E.P. 157,515, 6.10.19.

VARIOUS claims are made relating to the utilisation of cod and other fish; dried food products are prepared from the trimmed heads and other rejected parts, and fodder and manure from other offal.

—J. H. L.

*Baking powder or self-raising flour.* C. S. Brock. E.P. 157,581, 31.10.19 and 18.12.19.

POWDERED buttermilk is substituted for a part of the usual acid component of the baking powder or self-raising flour.—J. H. L.

*Milk, blood, etc.; Conversion of dissolved, suspended, or molten substances — into the form of a fine powder.* E. Trutzer. G.P. 325,396, 11.6.18.

IN the desiccation of liquids, with or without chemical transformation, by atomising in a current of drying gases, the liquid is delivered on to the surface of a horizontal, rapidly rotating cylinder, from which it is thrown off tangentially in a finely divided form.—J. H. L.



*Vegetables, vegetable refuse, and the like; Production of extracts or worts from fresh* — M. Trüstedt. G.P. 326,178, 3.3.18.

THE material is subjected for a short time to the action of steam under about 3 atm. pressure, in an autoclave, so that the tissue cells are opened and the juice from the material is cooled rapidly, and gradually released from pressure whilst cooling. In this way the aroma of the material is retained. —J. H. L.

*Yeast; Production of a foodstuff rich in protein from* — F. Konther. G.P. 328,260, 18.1.18.

SUGAR and yeast are introduced into boiling water, and the product is evaporated to dryness and heated to 130° C. or a higher temperature. To avoid evaporation as far as possible, the sugar may be melted with 10–15% of water and mixed with 10–60% of yeast and malt embryos (malt combs), the mass then being dried and heated. The product may be employed for the preparation of jam or beverages, or as a cacao substitute. —J. H. L.

*Food preparation from fresh blood; Manufacture of a* — F. Sgalitzer. E.P. 143,919, 28.5.20. Conv., 15.1.16.

SEE G.P. 296,925 of 1916; J., 1917, 665.

*Viscous liquids [sweetened condensed milk]; Method of treating* — R. Rafn. U.S.P. 1,368,291, 15.2.21. Appl., 15.10.18.

SEE E.P. 121,117 of 1918; J., 1919, 596 A.

*Semolina; Treatment of* — A. R. Goodwin. U.S.P. 1,368,792, 15.2.21. Appl., 30.10.18.

SEE E.P. 121,408 of 1918; J., 1919, 89 A.

*Compounding fats and oils.* E.P. 158,175. See XII.

*Coconuts.* U.S.P. 1,366,338—9. See XII.

*Milk sugar.* U.S.P. 1,366,822. See XVII.

*Destroying cellular structures.* U.S.P. 1,355,476. See XIXb.

### XIXb.—WATER PURIFICATION; SANITATION.

*Water; Detection of nitrites and nitrates in* — Escaich. Soc. Pharm. Paris, 7.7.20. Ann. Chim. Analyt., 1921, 3, 56—57.

FIFTEEN c.c. of the water is treated with 2 c.c. of 10% antipyrine solution and 4 drops of acid mercuric sulphate solution, and a drop of potassium ferricyanide solution is added; a red coloration develops if the water contains as little as 0.1 mg. of nitrites per litre. The test may be applied to the detection of nitrates after these have been reduced to nitrite by means of amalgamated aluminium. If a large amount of chloride is present, the water must be treated with silver nitrate and filtered before the test for nitrites is applied; in testing for nitrates, the treatment with silver nitrate is made after the nitrates have been reduced. —W. P. S.

*Hardness [of water]; Soap solutions for determination of* — A. Krieger. Chem.-Zeit., 1921, 45, 172—173.

FOR hardness determinations the author recommends the use of soap solution of 10 times the concentration of the usual Clark's solution. The burette should be graduated in degrees of hardness, and a 50 c.c. bottle should be used for the sample. —W. J. W.

*Carbon monoxide.* Florentin and Vandenberghe. See II. A.

*Chlorine in air.* Matignon. See VII.

*Mustard gas and thiodiglycol.* Grignard and others. See XX.

#### PATENTS.

*Filter [; Water —, with de-gassing device].* W. Hunold. G.P. 323,720, 6.11.18.

AIR is prevented from entering the filter bed along with the feed water by a de-aerating device operated automatically by a float in the supply pipe. The latter is closed at the top and extends down into or below the filtering material, the lower end being open or pierced with holes. A cock suitably disposed in the upper part of the supply pipe permits of the air collected therein being removed. The feed water passes over the float valve, and, becoming thereby atomised, oxidation of iron and similar substances contained in solution in the water is effected by the air collected in the upper part of the supply tube. —J. S. G. T.

*Water; Manufacture of agents for removing iron and manganese from, and decolorising and clearing* — A. Braedt. G.P. 328,630, 5.9.18.

MANGANESE dioxide is precipitated along with finely divided silicates, or is mixed with the same in a fine state of division and the mixture pressed, dried, and granulated. The hard fragments obtained are very efficient for removing iron and manganese from water. —C. I.

*Cellular structures; Method of destroying* — C. Hering. U.S.P. 1,355,476, 12.10.20. Appl., 27.1.15.

DISRUPTIVE changes in cellular structures immersed in a liquid are produced by subjecting the liquid to rapid successive positive and negative pressure changes. A strong vessel is filled with liquid in which the material under treatment is immersed. If this is liquid it is contained in a rubber bag or in a container closed by a rubber membrane. The pressure changes are produced mechanically by means of a plunger or piston. The process may be applied to the destruction of organisms in milk, water, etc., to the treatment of meat to improve its tenderness, to the disintegration of wood for the production of pulp, etc.

*Animal and vegetable pests; Material for destruction of* — R. Haberer und Co. G.P. 329,201, 2.8.18.

THE alkaloids and bitter principles derived from leguminous plants are suitable for the destruction of pests. The extract from lupins, on account of the gummy material which it contains, is especially suitable for spraying plants. —W. J. W.

*Gas from garbage.* U.S.P. 1,367,321. See IIa.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Morphine in opium and its preparations; Determination of* — T. Ugarte. J. Pharm. Chim., 1921, 23, 129—131.

ONE g. of the opium is heated for 5 mins. on a water bath with 10 c.c. of 67% alcohol, the solution filtered and the extraction repeated three times. The alcoholic extracts are mixed (in the case of tincture of opium the determination is commenced at this stage of the procedure), evaporated, the residue dried at 100° C. for 15 mins., and extracted with four successive quantities (5 c.c.) of cold water. The aqueous extracts are filtered, evaporated, the residue dissolved in 2 c.c. of water saturated previously with morphine, 3 c.c. of N/1 ammonia,

also saturated with morphine, is added, and the mixture is shaken with 10 c.c. of ether. After the addition of a further 20 c.c. of ether the mixture is set aside for 30 mins., the crystalline precipitate of morphine then collected, washed with water saturated with morphine, dried at 100° C., and weighed.—W. P. S.

*Meconic acid; Synthesis and constitution of* —. H. Thoms and R. Pietrulla. Ber. deuts. Pharm. Ges., 1921, 31, 4—19.

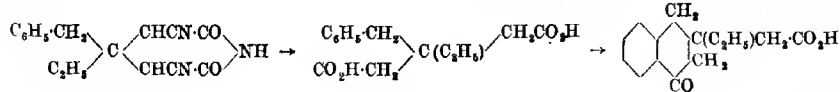
THE constitution of meconic acid as 3-hydroxy- $\gamma$ -pyrone-2,6-dicarboxylic acid, and its close relationship to chelidonic acid, was established by a synthesis of the acid from acetylhydioxalic ester. This substance on bromination in chloroform solution gave a monobromo-substitution product which immediately condensed with loss of water to monobromochelidonic acid (3-bromo- $\gamma$ -pyrone-2,6-dicarboxylic ester), forming colourless crystals from alcohol, m.p. 68° C. The substitution of hydroxyl for bromine in this compound without far-reaching decomposition presented great difficulty, but eventually a 35% yield of potassium meconate was obtained by the action of a slight excess of 5% potassium hydroxide on an acetone solution of the bromo-ester.—G. F. M.

*Humic acids; Constitution of* —. M. Popp. Brennstoff-Chem., 1920, 1, 58—59.

THE classification of humic acids according to Oden (Die Huminsäure, 1919) is given, with the addition of a new product—an acid obtained by P. Brat from peat—to which the name humalic acid is given. Its composition is C 43%, H 6%, O 51%; equivalent weight about 350, and m.p. 168° C. It is soluble in water and alcohol, and all its salts are soluble in water. The solubility of its calcium salt may be important in view of possible applications in medicine—as a substitute for calcium chloride, over which it possesses the advantage that it does not act as an irritant. In alkaline solution it gives a red coloration with ferric chloride, but no precipitate. It behaves as an aldehyde, and has a strongly reducing action.—H. J. H.

*Tetrahydronaphthalene; Formation of derivatives of* — from  $\gamma$ -phenyl fatty acids. G. A. R. Kon and A. Stevenson. Chem. Soc. Trans., 1921, 119, 87—94.

ALTHOUGH  $\gamma$ -phenylbutyric acid itself apparently does not undergo condensation to tetrahydronaphthalene, the ease with which the ring closes seems to be influenced by the character of the groups attached to the  $\beta$ -position of the side chain, and the hydrolysis of the condensation product obtained by Guareschi's method (Atti. R. Accad. Sci. Torino, 1900—1901, 36, 443) from benzyl ethyl ketone, ethyl cyanoacetate, and ammonia does not give the acid that would have been expected, but the tetrahydronaphthalene derivative formed from it by ring formation, according to the following scheme:—



—G. F. M.

*Chloropicrin; Preparation of* — from picric acid and trinitrotoluenes. K. J. P. Orton and P. V. McKie. Chem. Soc. Trans., 1921, 119, 29—33.

By passing chlorine into a cooled suspension of picric acid (sodium picrate) in aqueous sodium carbonate, a yield of 200% of chloropicrin may be obtained (E.P. 142,878; J., 1920, 527 A); 15—17 equivalents of alkali give the best results. In

concentrated sodium hydroxide the yield is much diminished, but with 13 equivalents of sodium hydroxide, of lower concentration than N/10, good results may be secured. Low temperature is favourable to the reaction; presence of sodium chloride retards it. By the action of hypochlorite solution on *o*- and *p*-mononitrophenol yields of only 10% and 33—34%, respectively, are obtained. With 2,4-dinitrophenol, 50% is reached. For the preparation of chloropicrin by the action of hypochlorite on trinitrotoluene and T.N.T. residues the best results are obtained by sludging the nitro-compound with water and bleaching powder, the proportions being: nitro-compound, 1 pt.; bleaching powder, 15 pts.; and water, 25 pts. The mixture is gradually heated, and steam is passed through. Grades I. and II. of trinitrotoluene give 82% and 84% yields; T.N.T. residues yield 70%. With dinitrotoluene and *s*-trinitrobenzene 4% and 53%, respectively, are obtained.—W. J. W.

*$\beta\beta$ -Di-iodoethyl sulphide and its application to the detection and estimation of yperite (dichloroethyl sulphide; mustard gas).* [Determination of thiodiglycol.] V. Grignard, G. Rivat, and G. Scatchard. Ann. Chim., 1921, 15, 5—18.

YPERITE ( $\beta\beta$ -dichloroethyl sulphide) reacts with hydriodic acid to give  $\beta\beta$ -di-iodoethyl sulphide, m.p. 62° C., and the ready formation of this compound is made the basis of a method for the detection and estimation of yperite. 5 c.c. of 54% hydriodic acid and 15 c.c. of acetic acid are heated for 15 mins. at 70° C. under an air condenser, and the mixture is then cooled and made up to 500 c.c. In 50 c.c. of this solution the iodine is liberated by the addition of 10 c.c. of a 10% solution of sodium nitrite, the iodine is extracted with carbon tetrachloride, and the extract is washed with water and then titrated with N/10 thiosulphate, A, c.c. being required. A second operation is carried out, about 1 g. of yperite being heated this time with the hydriodic and acetic acids, but before making the volume up to 500 c.c., 100 c.c. of carbon tetrachloride is added. The mixture is vigorously shaken, the two layers are allowed to separate, and from the aqueous layer (400 c.c.) 50 c.c. is taken and titrated with N/10 thiosulphate, A<sub>1</sub> c.c. being required. The carbon tetrachloride layer is then carefully separated and the free iodine in it is titrated with N/10 thiosulphate, A<sub>2</sub> c.c. being required. Then if the actual weight of yperite taken was P g., the sample contains  $(10A_1 + 8.5 - 8A_2 - A_2) \cdot 0.82 / P\%$  of yperite. For the detection of yperite a solution containing 20 g. of sodium iodide, 40 drops of a 7.5% solution of copper sulphate, and 2 c.c. of a 35% solution of gum arabic in 200 c.c. is used. To 4 c.c. of a yperite solution 1 c.c. of this reagent is added and a turbidity is produced after an interval of time varying with the temperature and the concentration of the yperite solution. In this way it is possible to detect 7 pts. of yperite in 1,000,000 of air by bubbling 3—4 l. of air through the reagent.

The formation of di-iodoethyl sulphide may also be made use of in the estimation of thiodiglycol. 0.7—0.8 g. of this substance is weighed out into a 50 c.c. conical flask, 5 c.c. of 54% hydriodic acid is added and the mixture is heated at 70°—75° C. for 15—20 mins. The mixture is cooled, filtered through glass wool, and the precipitate rapidly washed with cold water. In the filtrate the free iodine is esti-

mated by titration with  $N/10$  thiosulphate and then the acidity by titration with  $N/1$  sodium hydroxide, using phenolphthalein as indicator. If these two titrations give B c.c. and A c.c. respectively, and a blank estimation without any thiodiglycol gives  $B_0$  and  $A_0$  c.c., then the weight of thiodiglycol in the sample weighed out is  $61[A_0 - A - (B - B_0)/20]$  mg.—W. G.

*Iodoamidines.* J. Bougault und P. Rohin. *Comptes rend.*, 1921, 172, 452—454.

CONFIRMATION of the view that the iodoamidines are derivatives of hypoiodous acid is given by the fact that iodobenzamidine reacts quantitatively with antipyrine, vanillin, and thymol respectively to give their iodo derivatives. With acetic anhydride it gives a compound,  $C_8H_8N_4I_2(CH_3CO)_2O$ , which is apparently a derivative of a di-iodoamidine.—W. G.

*Tartaric acid; Decomposition of — by heat.* F. D. Chattaway and F. E. Ray. *Chem. Soc. Trans.*, 1921, 119, 34—37.

If tartaric acid is heated under reduced pressure so as to effect decomposition at the lowest temperature possible, decomposition takes place in two stages and without any side-reactions. In the first stage only water is liberated, and a residue of the nature of a lactide remains. On further heating, this decomposes into carbon monoxide and dioxide, and formic, acetic, and pyruvic acids. The average amounts of decomposition products from 1 g.-mol. of tartaric acid were: water, 18.8 g.; formic acid, 2.4; acetic acid, 4.9; pyruvic acid, 1.1; carbon monoxide, 21.3; and carbon dioxide, 43 g.

—W. J. W.

*Esterification by zirconium oxide.* A. Mailhe and F. de Godon. *Bull. Soc. Chim.*, 1921, 29, 101—106.

ZIRCONIUM oxide is a good catalyst for the preparation of esters of aliphatic acids by passing the mixed vapours of the acid and alcohol over the oxide at  $270^\circ$ — $290^\circ$  C. The yield of ester depends on the weight of oxide used, the velocity of flow of the vapours, and the proportion of acid to alcohol.

—W. G.

*Alcohols; Dehydrogenation of — by catalytic oxidation.* C. Moureu and G. Mignouac. *Bull. Soc. Chim.*, 1921, 29, 88—101.

A DETAILED account of work already published (cf. J., 1920, 247 A, 764 A).—W. G.

*Amines; New method of preparation of secondary — and attempts to alkylate these bases.* A. Mailhe. *Bull. Soc. Chim.*, 1921, 29, 106—110.

A DETAILED account of work already published (cf. J., 1921, 194 A).—W. G.

*Organic compounds; Method of determining and verifying the purity of — by oxidation with chromic acid.* H. Cordebar. *Ann. Chim. Anal.*, 1921, 3, 49—53.

OXIDATION by heating with an excess of standard bichromate solution and sulphuric acid (as in the method for the determination of glycerol) and subsequent titration of the excess of bichromate with ammonium ferrous sulphate solution affords a means of determining the quantity or purity of a considerable variety of organic compounds.

—W. P. S.

*Cineol; The cresineol method for the determination of —.* C. E. Sage and J. D. Kettle. *Perf. Essent. Oil Rec.*, 1921, 12, 44—46.

For the determination of cineol in encalyptus oils, the cresineol method (J., 1920 A, 610 A) yields

slightly higher figures than does the phosphoric acid method. When the cineol phosphate is weighed, the factor 0.588 should be used in calculating the amount of cineol present, and not the theoretical factor 0.611.—W. P. S.

*Cineol; The cresineol method for the determination of —.* T. T. Cocking. *Perf. Essent. Oil Rec.*, 1921, 12, 44.

It is pointed out that as long as the phosphoric acid method of determining cineol is official, an oil which gives 55% of cineol as determined by the cresineol method (J., 1920, 610 A) will not conform to the requirements of the British Pharmacopoeia.

—W. P. S.

*Salicinase.* Bertrand and Compton. See XVIII

*Iodoform reaction.* Schoorl. See XXIII.

#### PATENTS.

*Anæsthetic compounds.* O. Kamm, R. Adams, and E. H. Volwiler, Assrs. to The Abbott Laboratories. U.S.P. (A) 1,358,750 and (B) 1,358,751 16.11.20. Appl., 23.1.20.

(A) THE di-*n*-butylaminoalkyl esters of aromatic acids, of the general formula,  $Ar.CO.O.(CH_2)_nNR$  ( $Ar$  is an aryl group containing a benzene nucleus and  $R$  is an *n*-butyl group), are obtained by the interaction of benzoyl chloride or a substitution product and di-*n*-butylaminoethyl alcohol. The latter, which has not previously been described, is prepared by the interaction of ethylenechlorhydrin and di-*n*-butylamine; it boils at  $225^\circ$ — $230^\circ$  C. with slight decomposition. The hydrochlorides of the  $\beta$ -di-*n*-butylaminoethyl and  $\gamma$ -di-*n*-butylamino propyl esters of *p*-aminobenzoic acid melt at  $170^\circ$ — $172^\circ$  and  $104^\circ$ — $105^\circ$  C. respectively. (B) The  $\gamma$ -dialkylamino-*n*-propyl esters of aromatic acids of the general formula,  $Ar.CO.O.(CH_2)_nNR'R''$  ( $Ar$  is an aryl group containing a benzene nucleus and  $R'$  and  $R''$  are alkyl groups, one of which is large than an ethyl group), are prepared by the interaction of benzoyl chloride or a substitution product and  $\gamma$ -dialkylaminopropyl alcohol.  $\gamma$ -Di-*n*-butylaminopropyl alcohol, b.p.  $235^\circ$ — $240^\circ$  C. (slight decomp.),  $170^\circ$  C. at 80 mm., is obtained by the interaction of trimethylenechlorhydrin and di-*n*-butylamine. The hydrochlorides of the  $\gamma$ -di-*n*-butylaminopropyl, di-isobutylaminopropyl, di-isocamylaminopropyl, and di-isopropylaminopropyl esters of *p*-aminobenzoic acid melt at  $104^\circ$ — $105^\circ$ ,  $169^\circ$ — $170^\circ$ , and  $178^\circ$ — $179^\circ$  C. respectively.

*Urea; Manufacture of — from cyanamide with the aid of solid catalysts.* Akt.-Ges. für Stickstoffdünger. G.P. 301,263, 25.3.16.

REDUCING substances which would contaminate the catalysts are eliminated or rendered harmless before the reaction. The reducing substances are chiefly sulphur compounds coming from the crude calcium cyanamide and from the gases containing carbon dioxide used in preparing the cyanamide solution. Permanganates, persulphates and hydrogen peroxide may be used for the removal of the reducing substances.—C. A. C.

*Rhamnus carulea; Manufacture of an extract from the bark of — for therapeutic purposes.* Saccharin-Fabr. A.-G. vorm. Fahlberg, Lis and Co. G.P. 328,767, 25.8.18.

THE drug is heated with steam in an autoclave at  $140^\circ$  C., and the soluble substances are then extracted. By heating to  $140^\circ$  C. the therapeutically inactive substances insoluble in water and alcohol are hydrolysed and converted into soluble, active substances. The extract is milder and more efficacious than that prepared at  $100^\circ$  C.—C. A. C.

*Acetic anhydride; Process of making* — H. Dreyfus. U.S.P. 1,368,789, 15.2.21. Appl., 26.12.17.

SEE F.P. 478,951 of 1914; J., 1916, 1179.

*Non-flammable liquid.* E.P. 158,494. See I.

*Condensation products of naphthalene and its derivatives.* G.P. 301,713. See III.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Photographic plate; Action of light on the* —  
II. *The under-exposure period.* G. I. Higson.  
Phot. J., 1921, 61, 144—152.

IN extending the application of the equations previously given (J., 1921, 27 A, 99 A) to the under-exposure period of a plate characteristic curve it is deduced that films may be regarded as one grain in thickness. It is suggested that for most plates, and for exposures of over  $\frac{1}{125}$  sec. to white light, density in the under-exposure region is proportional to the square of the light intensity (for intensity scale exposures). The DI (density-intensity) curve has a point of inflexion and the intercept on the I axis of the tangent at this point,  $\lambda$ , is regarded as a constant of the plate;  $\lambda$  represents the speed of the plate for very short exposures. Conclusions as to "cut" are also deduced which are not quite in agreement with Renwick's conclusions (Phot. J., 1913, 53, 127). The ratio between inertia ( $i$ ) and  $\lambda$  is constant and equals 1.71; also for any one plate and under such conditions that  $D\lambda^2$ , the ratio between  $u$  (the slope of the above tangent) and  $\gamma$  (development factor) is proportional to the speed of the plate. Experimental details of the determination of  $\lambda$  are given and also some conclusions with respect to time-scale exposures in the under-exposure region.—B. V. S.

*Photographic plate; Blackening of a* — as a function of intensity of light and time of exposure.  
P. S. Helmick. Phys. Rev., 1921, 17, 135—146.

PLATES, specially coated on plate glass, were exposed for varying times, or at varying distances, in monochromatic light obtained by a Hilger Monochromatic Illuminator. Three types of plate and three wave-lengths of light were used. Plates were developed in a quinol developer for a standard time, a correction to this being applied for variation in temperature. A mathematical and a graphic method are given for evaluation of the constants in the equation relating density of image to exposure given. The methods outlined are used for calibrating plates which may subsequently be used for the photographic determination of light intensities. The conclusions of Kron and Lemon that Schwarzschild's constant  $p$ , in the equation  $I\lambda^p = C$  for equal density, varies with  $I$ , are confirmed and experimental values of  $p$  are given varying from 0.68 to 1.95.—B. V. S.

*Photographic research; Some new directions for* —  
J. Rheinberg. Phot. J., 1921, 61, 120—125.

THE author draws attention to the scope for research in a study of the alteration of the physical properties of colloids by the action of light and illustrates it by some recent processes dependent upon such alterations. To obtain a multicolour screen for a process of colour photography a collodion film is coated with a resist film containing green ferric ammonium citrate and uranium nitrate. The film is exposed behind a line screen, the exposed parts becoming permeable to acidified alcohol by means of which dyes may be extracted

from or introduced into the collodion film underlying the exposed parts. Gum arabic, fish-glue, and albumin may be used for the resist film; the process may be repeated by removing the film and recoating in the case of the two former, or by washing out the soluble salts and re-sensitising in the case of the albumin. In a process for the manufacture of graticules the resist film, of collodion, fish-glue, gum arabic or albumin, containing an iron salt and sensitised by alcoholic solution of silver nitrate, is rendered more permeable to hydrofluoric acid gas by exposure to light. Reference is also made to the fact that the tendency of collodion films to dry with a wavy surface is due to the very different rates of evaporation of the ether and spirit used as solvents, and can be overcome by replacing these by methyl alcohol.—B. V. S.

*Latent [photographic] image; Theory of the development of the* — M. Volmer. Z. wiss. Phot., 1921, 20, 189—198.

CAREFULLY prepared finely-divided silver was obtained by reduction of silver nitrate by formaldehyde. It was found that the presence of a small quantity of this in a solution of a developer increased the rate of oxidation when air was bubbled through the solution. The results are given for catechol, metol, quinol, glycin, *p*-aminophenol, amidol, and eikonogen, the effect being most marked with *p*-aminophenol and least with metol and eikonogen. Similar results were obtained with *p*-aminophenol and metallic gold and platinum, but no acceleration of oxidation was produced by glass powder, silver sand, or barium sulphate. The possible connexion between the catalytic effect of metallic silver in this experiment and ordinary development processes is discussed.—B. V. S.

*[Photographic] development; White light* —  
B. V. Storr. Phot. J., 1921 61, 126—128.

A SHORT account is given of the work of Lüppo-Cramer which has resulted in the process by which plates may be developed in white light after a preliminary treatment with a weak solution of certain dyes, chiefly those of the safranin class (J., 1921, 23 A, 99 A).—B. V. S.

*Persulphate [photographic] reducer; Irregularities in the action of* — A. and L. Lumière and A. Seyewetz. Brit. J. Phot., 1921, 68, 124—125.  
Phot. J., 1921, 61, 144.

THE chief cause of irregularity in the reducing action of persulphate is variation in acidity. The explanation previously advanced by Sheppard (J., 1913, 607 A) attributing the variation to variable iron content is shown to be insufficient, as active solutions have been made quite free from iron. The presence of chloride, either in the persulphate or in the tap-water used for dissolving it, may also produce irregularities, since 0.04 g. of sodium chloride per 100 c.c. of 4% persulphate solution completely inhibits the solvent action if the solution is only slightly acid; larger quantities of chloride lead to bleaching or to slight intensification due to change of colour if the acidity is increased also. Sulphates and nitrates have a slight retarding effect in slightly acid solutions which is overcome by increasing the acidity. Chemical reducers, such as thiosulphate left in the film by insufficient washing, reduce the persulphate and prevent its effect on the silver image.—B. V. S.

*Colloid chemistry and photography. LI. Staining as a reagent for [extent of] surface.* Lüppo-Cramer. Kolloid Zeits., 1921, 28, 90—93.

CONSIDERATION of the relative colour-sensitising capacities of silver haloid salts (iodide, bromide, and chloride) and their relative staining powers with various dyes, along with other similar factors,

e.g., the observed disintegration of silver bromide grains on conversion to iodide, leads to the general conclusion that the effective surface of the grains is much more important in determining degree of adsorption of dyes than is the purely chemical nature of the halide.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

*Explosives; Some properties of* —. R. Robertson. Chem. Soc. Trans., 1921, 119, 1—29.

WITH nitro-derivatives of the benzene series, the lowest heat of formation is found when the nitro-groups are adjacent; in such cases stability is lower, and the sensitiveness to impact increases. Thus 1,2,4-trinitrobenzene and 2,3,4-trinitrotoluene are inferior to their respective 1,3,5- and 2,4,6-isomerides. The heats of explosion of various explosives were determined by detonating them in a steel envelope at a loading density of 1.3; in this way trinitrotoluene gave 924, and picric acid 914 cal., as compared with 641 and 793 found by Poppenberg. The heat of explosion of dinitrobenzene is 820; this substance was used, without admixture, as a high explosive for shells during the war. In connexion with the decomposition of explosives at even low temperatures, many temperature coefficients have been determined; with nitroglycerin quantitative results have been obtained down to 60° C. The decreasing resistance to decomposition of a series of explosives was found to correspond to the ease with which they can be hydrolysed. With aromatic nitro-compounds, a measurable evolution of gas is only reached with the trinitro-derivative. Rate of detonation of explosives appears to fall in line with their heat of explosion and is therefore dependent on their molecular structure. In regard to amatols, their sensitiveness is not increased by heating, but is appreciably increased by presence of grit and of certain salts. The rate of detonation of trinitrotoluene is not much reduced by admixture with ammonium nitrate up to 40%, the respective figures being 6950 m.p.s. for trinitrotoluene (d. 1.57), and 6470 m.p.s. for 40/60 amatol; 80/20 amatol gives 4620 and 5080 m.p.s. for densities of 1.3 and 1.5 respectively; and this rate is not appreciably altered by varying the thickness of the container wall. (Cf. J., 1921, 26—27 R.)—W. J. W.

*Trinitrotolymethylnitroamine* [; Preparation of — from trinitrotoluenes]. O. L. Brady and W. H. Gibson. Chem. Soc. Trans., 1921, 119, 98—104.

By treatment of 2,3,4-trinitrotoluene in alcoholic solution with methylamine hydrochloride and ammonia solution, 2,4-dinitromethyl-*m*-toluidine is produced; 4,6-dinitromethyl-*m*-toluidine is similarly prepared from 3,4,6-trinitrotoluene. With dimethylamine hydrochloride, 2,4- and 4,6-dinitrodimethyl-*m*-toluidines are formed. For the preparation of 2,4,6-trinitrotolymethylnitroamine, 2,4- or 4,6-dinitromethyl- or dimethyl-*m*-toluidines, or a mixture of these obtained by the action of methylamine hydrochloride containing dimethylamine on a mixture of 2,3,4- and 3,4,6-trinitrotoluenes, is introduced into nitric acid (sp. gr. 1.5), warmed to 50° C., and the mixture is heated on a water-bath till evolution of nitrous fumes ceases, after which it is cooled and water added, the trinitrotolymethylnitroamine separating out as an oil which solidifies on standing. The solid is recrystallised from alcohol containing a few drops of acetic acid, or from a mixture of alcohol and benzene. Other compounds of which the preparation is described are: 2,4- and 4,6-dinitrotolymethylnitroamine, 2,4,6-trinitrotolymethylnitro-

amine, 2,4- and 4,6-dinitrotolymethylnitrosoamine and 2,4,6-trinitromethyl-*m*-toluidine.

—W. J. W.

*Chloropicrin*. Orton and McKie. See XX.

### PATENTS.

*Nitrocellulose powder; Coated — and process of making the same*. R. G. Woodbridge, jun., Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,354,640, 5.10.20. Appl., 20.2.20.

NITROCELLULOSE powder (100 pts.) is mixed with about 2 pts. of a non-volatile solvent, e.g., dinitrotoluene, in a revolving drum, 1—10 pts. of black powder is added, and agitation is continued at a temperature slightly above the melting point of the dinitrotoluene, so as to obtain nitrocellulose powder, the grains of which have a firmly adherent coating of black powder.

*Potassium chlorate; Form of — and process of producing the same*. E. R. Wolcott, Assr. to International Precipitation Co. U.S.P. 1,355,203, 12.10.20. Appl., 17.3.19.

POTASSIUM chlorate in the form of silky or fibrous crystals, specially suitable for use in explosives, as they are less liable than the ordinary form to produce explosions on grinding with easily combustible substances, is obtained by boiling a saturated solution of potassium chlorate with an aqueous solution of the tarry product formed by treating Californian crude petroleum with oleum, which is probably composed of sulphonated products of unsaturated hydrocarbons; after boiling, the mixture is filtered and the solution allowed to crystallise.

*Screens [used in nitrocellulose manufacture]; Process of cleaning —*. R. G. Woodbridge, jun., Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,367,111, 1.2.21. Appl., 31.5.17.

METALLIC screens through which nitrocellulose has been passed are cleansed by the application of an alkaline salt solution, the action being controlled by regulation of temperature and concentration.

—W. J. W.

*Explosive powder*. A. Langmeier, Assr. to Hercules Powder Co. U.S.P. 1,367,608, 8.2.21. Appl., 27.3.20.

AN explosive consists of trinitrotoluene mixed with ammonium perchlorate and sodium nitrate, these salts being first coated with trinitrotoluene.

—W. J. W.

*Match-heads; Composition for —*. W. A. Fairburn, Assr. to The Diamond Match Co. Reissue 15,039, 15.2.21, of U.S.P. 1,360,283, 30.11.20. Appl., 3.1.21.

SEE J., 1921, 101 A.

## XXIII.—ANALYSIS.

*Anemometer; A new —*. H. Gerdien and R. Holm. Wiss. Veröffentl. Siemens-Konzern, 1920, 1, 107—121. Chem. Zentr., 1921, 92, 11, 251—253.

THE anemometer is constituted of a Wheatstone bridge, two arms of which are formed of very thin wires of high temperature coefficient, heated by an electric current and inserted directly behind one another in the stream. The leading wire is cooled by the stream to a greater extent than the second, and the galvanometer deflection thereby produced serves to indicate the magnitude of the velocity of the stream (cf. J., 1918, 167 r; 1920,

803 A). The apparatus is applicable to the measurement of the velocities of gases in pipes. If an obstruction is placed in the pipe, with one arm of the anemometer on either side of it, measurements of large velocities may be made. The only important correction necessary to the indications of the instrument is that due to alteration of temperature of the gas. The necessary correction can be calculated or mechanically compensated, the latter method eliminating any necessary correction to within 0.1% per degree centigrade. The device can also be employed for the measurement of high velocities by by-passing a constant fraction of the stream over the heated wires. The temperature of the wires is much below that necessary to cause corrosion of the wires due to combustion of dust or to cause ignition of explosive gaseous mixtures. Impurities in the gas influence the readings of the instrument. Thus 1% of illuminating gas admixed with air reduces the sensitiveness of the instrument by about 35%.—J. S. G. T.

*Chlorine and bromine ions; Detection of — in the presence of iodine ions.* E. Ludwig. *Bul. Soc. Chim. România*, 1920, 2, 23–28.

The halogens are precipitated from boiling acid solution with silver nitrate, the precipitate being collected and washed free from silver nitrate. It is then boiled with as small an amount as possible of N/10 ammonia solution. To a portion of the liquid a little sodium chloride is added. A turbidity indicates chlorides in the original mixture. To another portion of the liquid one drop of a concentrated solution of sodium bromide is added. A turbidity indicates bromides or a trace of chlorides in the original mixture. To confirm the bromides the residue left after treating the precipitate with N/10 ammonia is boiled with N/1 ammonia solution. The solution obtained is warmed with zinc and an excess of sulphuric acid. When the action is complete the liquid is decanted and tested for bromides by the addition of chlorine water in the presence of chloroform in the usual manner.—W. G.

*Acids or bases; Volumetric determination of mixtures of —, and of polybasic acids or bases.* H. T. Tizard and A. R. Boerce. *Chem. Soc. Trans.*, 1921, 119, 132–142.

MATHEMATICAL expressions are developed to elucidate the conditions which control the accuracy of titration of mixtures of acids or of bases, or of di- or tri-valent acids or bases, which is merely a special case of the former. In a solution containing an equivalent of a weak acid  $HA_x$  and  $x$  equivalents of a weak acid  $HA_y$ , the dissociation constants of which are  $K_1$  and  $K_2$  respectively, the indicator suitable for the titration will be conditioned by the hydron concentration,  $C_H$ , at the equivalent point. It is shown that  $C_H$  (or  $C_{OH}$  in the case of bases) =  $\sqrt{xK_1K_2}$ , and the condition of titration of the stronger acid (or base) to 1% is that  $K_1/xK_2 > 2.5 \times 10^4$ , and for titration to 0.1%  $K_1/xK_2 > 2.5 \times 10^5$ . These general conditions can be applied in nearly every case except when solutions are so concentrated that the "neutral salt" effect comes into play. The titration of a single acid or base in presence of water is simply a special case where the second dissociation constant  $K_2$  becomes  $K \times V$  where  $V$  is the dilution and  $K_w$  the so-called dissociation constant of water. Here the condition for titration to 1% works out to approximately  $K_1 = 2.5 \times 10^4$ . The application of these principles to the titration of ammonia, ammonia and aniline, ammonia and  $\beta$ -picoline, phenol and acetic acid, and acetic and chloroacetic acids is discussed.—G. F. M.

*Hydriodic acid; Electro-titration of — and its use as a standard in oxidimetry.* W. S. Hendrixson. *J. Amer. Chem. Soc.*, 1921, 43, 14–23.

Iodides may be accurately titrated electrometrically with potassium permanganate in sulphuric acid solution. The reaction is carried out in a three-necked bottle, which carries the burette, a tube for leading in carbon dioxide or air to agitate the solution, and a bright platinum electrode and the connecting tube of a calomel electrode. The EMF of the system is determined as the titration proceeds and the point where there is a sudden rise of EMF is the end point of the reaction. The reaction is affected by the presence of bromides and chlorides, but may be still carried out if the amount of chloride does not exceed that of the iodide and if the bromide concentration is not more than 25% of that of the iodide. Bichromate and iodate in N/50 and N/20 solutions may be accurately titrated by adding solutions of either to an excess of an iodide in sulphuric acid and titrating the excess by potassium permanganate.—J. F. S.

*Sulphuric acid; Volumetric determination of —.* C. Pezzi. *Giorn. Chim. Ind. Appl.*, 1921, 3, 10–11.

The presence of ferric salts, which tenaciously retain occluded benzidine sulphate, renders invalid Raschig's method of estimating sulphuric acid volumetrically by precipitating it as benzidine sulphate and titrating the latter, suspended in water, with N/10 sodium hydroxide solution at 50° C. The author finds that results accurate to within about 0.2% may be obtained, even when ferric salts are present, by titration of the precipitated benzidine sulphate with standard sodium nitrite solution. Two g. of benzidine is dissolved in 750 c.c. of water containing 3 c.c. of hydrochloric acid, the solution, when clear, being made up to 1 litre; 0.1 g.  $H_2SO_4$  requires 150 c.c. of this reagent, which is added, in the cold and with continual stirring, to the sulphate solution containing hydrochloric acid. The precipitate is allowed to settle, and the mother-liquor then filtered in small quantities through a double filter-paper on a Buchner funnel; the precipitate is afterwards brought on to the funnel, washed first with the mother-liquor, and finally several times with hot water, using 15 c.c. in all, and then rinsed off the paper into a beaker. The papers are treated in a small beaker with 15 c.c. of concentrated hydrochloric acid until completely destroyed, this solution being added to the suspension of the precipitate, which should be finely divided and quite free from clots. The whole liquid is then titrated with N/20 sodium nitrite solution at 10°–12° C. until, even 15 mins. after the titration is complete, a drop of the liquid colours potassium iodide-starch paper; 1 c.c. N/20 sodium nitrite is equivalent to 0.002452 g.  $H_2SO_4$ .—T. H. P.

See also pages (A) 205, *Coal* (Gray and King). 206, *Carbon monoxide* (Florentin and Vandenberghe); *Oil shale* (Lomax and Remfrey). 207, *Sulphur in benzene* (Jackson and Richardson). 209, *Tar oils* (Lazar); *Phenol* (Rodillon). 213, *Sulphide dye baths* (Herbig). 215, *Chlorine in air* (Matignon). 222, *Spongy platinum* (Feulgen). 223, *Thermo-elements* (Pfleiderer, also Fischer and Pfeleiderer). 226, *Tung oil* (Gardner); *Sulphonated oils* (Hart, also Bumcke). 230, *Bating materials* (Thomas); *Pumping power of tannery solutions* (Clafflin). 231, *Chrome-tanned leather* (Hou); *Pyroxylin compositions* (Lorenz); *Soil reaction* (Fisher). 232, *Phosphate in soils* (Shedd); *Superphosphate* (Müller). 233, *Sucrose, dextrose, and levulose* (Vosburgh); *Cuprous oxide* (Sarma). 235, *Lactose in milk* (Adriano); *Swelling of colloids* (Lüers and Schneider). 236, *Nitrates and nitrites*

in water (Escaich); *Hardness of water* (Krieger); *Morphine* (Ugarte). 237, *Mustard gas and thiodiglycol* (Grignard and others). 238, *Organic compounds* (Cordehard); *Cineol* (Sage and Kettle, also Cocking).

## PATENTS.

Membrane filters. G.P. 329,060 and 329,117. See I.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

## I.—GENERAL; PLANT; MACHINERY.

## APPLICATIONS.

Bäckström and Cederberg. Apparatus for carrying out highly exothermic catalytic reactions between gases. 8090. Mar. 15.  
Bale. Kiln. 9101. Mar. 24.  
Ballard. Furnaces. 8846. Mar. 22.  
Barber. Means for recovering colloidal matter from liquids. 8809. Mar. 22.  
Bouvery and Conort. Self-lubricating anti-friction products. 8534. Mar. 18. (Fr., 26.3.20.)  
Broadley. Crushing machines etc. 7623. Mar. 10.  
Davis. Evaporators, feed-water heaters, etc. 8572. Mar. 19.  
Duffield and Longbottom. Rotary furnaces. 7564. Mar. 9.  
Fasting. Process for drying material to be ground in revolving drums. 8023. Mar. 14. (Denmark, 22.3.20.)  
Fooks. 7664. See II.  
Gensecke, and Metallhank u. Metallurgische Ges. Utilisation of waste heat. 7886. Mar. 12.  
Henshilwood and Wood. Non-conducting material for steam boilers etc. 8027. Mar. 15.  
Heylandt Ges., and Unruh. Cooling and liquefying air etc. 7818. Mar. 11. (Ger., 28.7.20.)  
Mason. 8324. See II.  
Mauss. Heat treatment of liquid. 7454. Mar. 8.  
Mauss. Treating liquids with gases. 7455. Mar. 8.  
Mauss. Vacuum separation of colloidal matter from liquid mixtures. 7457. Mar. 8.  
Mauss. Centrifugal decanters. 7458. Mar. 8.  
Petsch and Still. Distillation columns. 8150. Mar. 15.  
Petzel. Bodies for filling columns, towers, etc. through which gas is passed in an opposite direction to liquid. 8274. Mar. 16. (Switz., 17.3.20.)  
Steigner. Kilns. 8303. Mar. 16.  
Tesla. Production of high vacua. 9098. Mar. 24.  
Torrance, and Torrance and Sons. Grinding-mills. 7770. Mar. 11.  
Whitfield. Drying-apparatus. 8966. Mar. 23.  
Winzer. Continuous-muffle furnaces or kilns. 8344. Mar. 17.  
Withers (Deuts. Evaporator A.-G.). Utilising heat contained in fuel residues of furnaces. 7317. Mar. 7.

## COMPLETE SPECIFICATIONS ACCEPTED.

10,174 (1919). Heeley. Distillation furnaces. (159,908.) Mar. 23.  
24,721 (1919). British Oxygen Co., and others. See X.  
26,045 (1919). De Haen Chem. Fahr. List. Method of making filter elements. (134,228.) Mar. 16.

27,466 (1919). MacLachlan, Stewart, and Clark. Boiling-pans and mixing-machines. (159,924.) Mar. 23.  
27,522 (1919). Seymour. Pulverising or fine-grinding. (159,925.) Mar. 23.  
30,502 (1919). Nitro-Fixation Synd., and Jenkins. See X.  
31,324 (1919). Imbery. Furnaces. (160,247.) Mar. 31.  
1453 (1920). Beyer. Ring furnace and drying plants. (160,328.) Mar. 31.  
7648 (1920). Blanch. Separating soluble from insoluble matter. (160,081.) Mar. 23.  
16,113 (1920). Elektro-Osmose A.-G. Leaching vegetable, animal, or mineral matter. (145,045.) Mar. 16.  
17,759 (1920). Cramm. Grinding or crushing mills. (145,599.) Mar. 23.  
20,008 (1920). Balthasar. Jets for evaporating liquids. (147,953.) Mar. 16.  
20,078 (1920). Schranz. Concentrating-apparatus for separating substances according to their specific gravity. (148,168.) Mar. 16.  
20,631 (1920). Danhardt. Filter for dry-cleaning gases and vapours. (148,800.) Mar. 23.

## II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

## APPLICATIONS.

Alexander. Kilns for drying and distilling fuels etc. 8144. Mar. 15.  
Bates. Fuel. 8164—8171. Mar. 16.  
Corthesy. Extracting gases from hydrocarbons etc. 8269. Mar. 16.  
Cutler and Meade. Gas-producers. 9210. Mar. 21.  
Davidson. Retorts for distilling coal etc. 8723. Mar. 21.  
Ferguson and Highett. Producing and utilising combustible gases for heating. 9177. Mar. 24.  
Fooks. Heat-treating bodies in a retort etc. 7664. Mar. 10.  
Ginet. Treating bituminous shales. 7557. Mar. 9.  
Glover, West, and West's Gas Improvement Co. Settings for vertical retorts. 7563. Mar. 9.  
Goold (Universal Oil Products Co.). Cracked petroleum oils and process of producing same. 7747. Mar. 11.  
Helps. Gas manufacture. 8631. Mar. 19.  
Hird. Apparatus for carbonising coal, shale, etc. 8636. Mar. 21.  
Illingworth. Production of smokeless fuels and coke. 9129. Mar. 24.  
Kimber and Walker. Destructive distillation of shale etc. 8268. Mar. 16.  
McDonald. Manufacture of gas. 7407. Mar. 8. (U.S., 10.3.20.)  
Marks (Stephens Engineering Co.). Processes of oxidising fuel. 7897. Mar. 12.  
Mason. Retort internally heated by gaseous fuel. 8324. Mar. 17.  
Morgan, and Thermal Industrial and Chemical Research Co. Treatment of peat etc. 7883. Mar. 12.  
Murray. Plant for compressing peat. 7312. Mar. 7.  
Murray. Gas-producers using peat. 7313. Mar. 7.  
Nesfield. Desulphurising oils. 9157. Mar. 24.  
Parrish, and South Metropolitan Gas Co. Utilising spent oxide from gas works etc. 7588. Mar. 9.  
Parker. Gas-producers. 8804. Mar. 22.  
Paterson, Smith, and Tulloch. Gas-producers. 8220. Mar. 16.  
Planson and Vielle. Manufacture of hydrocarbons from coal etc. 9053. Mar. 24. (Ger., 24.3.20.)



Preston. Recovery of ammonium compounds from coal etc. 7237. Mar. 7.  
 Rigby. Treatment of peat. 8539. Mar. 19.  
 Rollason. Manufacture of water-gas. 7248. Mar. 7.  
 Withers. 7317. *See I.*

## COMPLETE SPECIFICATIONS ACCEPTED.

16,364 (1915). Planiauerke A.-G. Arc-lamp electrodes. Mar. 16.  
 13,638 (1917). Felt. Purification of illuminating and other gases. (159,905.) Mar. 23.  
 15,756 (1918). Soc. Indus. de Prod. Chimiques. Recovery of ammonia from coke-oven and like gases. (127,549.) Mar. 23.  
 10,174 (1919). Heeley. *See I.*  
 24,605 (1919). Holes and Manley. Cracking of hydrocarbon oils. (160,200.) Mar. 31.  
 24,804 (1919). Gratz. Gas manufacture. (160,202.) Mar. 31.  
 29,491 (1919). Fleming. Condensing and treating distillates from oil-cracking stills. (159,574.) Mar. 16.  
 31,074 (1919). Davidson. Preparation of peat fuel for producer or other gas. (159,996.) Mar. 23.  
 31,138 (1919). Goold (Universal Oil Products Co.). Converting heavy into lighter hydrocarbons. (160,236.) Mar. 31.  
 31,820 (1919). Dale. Coal and like briquettes. (160,279.) Mar. 31.  
 1392 (1920). South Metropolitan Gas Co., and Gair. Incandescent gas mantles. (160,040.) Mar. 23.  
 1550 (1920). Leadbeater. Manufacture of artificial fuel. (160,042.) Mar. 23.  
 4232 (1920). Bourdon. Gas-purifying apparatus. (139,758.) Mar. 16.  
 7783 (1920). Hislop. Fuel. (160,083.) Mar. 23.  
 10,446 (1920). Jackson (Internat. Gasoline Process Corp.). Apparatus for distilling oils or other fluids. (159,774.) Mar. 16.  
 16,905 (1920). Farb. v. m. Meister, Lucius, u. Brüning. Manufacture of methane. (146,114.) Mar. 16.  
 19,202 (1920). Oswald and Brown. Apparatus for extracting oil from oleiferous sandstone shale etc. (160,114.) Mar. 23.

## III.—TAR AND TAR PRODUCTS.

## APPLICATIONS.

Adam, Lewcock, Galbraith, and Siderfin. Production of aminophenols etc. 8604. Mar. 19.  
 Adam, Galbraith, Siderfin, and Tallantyre. Plastic material, and process of preparing same from tar distillates. 8605. Mar. 19.  
 Benn, Benn, and Benn. Tar distillation etc. stills. 8432. Mar. 18.  
 Caspari and Warburton. Manufacture of benzene monosulphonic acid. 7830. Mar. 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

18,943 (1920). Meister, Lucius, u. Brüning. Manufacture of pyridine bases. (147,000.) Mar. 23.  
 23,569 (1920). Anderson and Meikle. Treating mineral tars other than coal tar. (159,802.) Mar. 16.

## IV.—COLOURING MATTERS AND DYES.

## APPLICATION.

Imray (Soc. Chem. Ind. Basle). Manufacture of chromiferous complex organic compounds and of chromiferous azo dyestuffs therefrom. 8516. Mar. 18.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

## APPLICATIONS.

British Cellulose and Chem. Manuf. Co., and Lely. Apparatus for spinning artificial threads etc. 7771. Mar. 11.  
 Carpmal (Bagley and Sewell Co.). Manufacture of paper. 8282, 8394, 8395. Mar. 16 and 17.  
 Dreyfus. Manufacture of solutions or preparations having a basis of cellulose derivatives. 8684. Mar. 21.  
 Dreyfus. Manufacture of products from cellulose derivatives. 8685. Mar. 21.  
 Fort, Mackenzie, and Robinson. Treatment and purification of yarns and fabrics composed of vegetable fibres. 8069. Mar. 15.  
 Green. Compositions of cellulose acetate and nitrate. 8348. Mar. 17.  
 Kershaw and Zdanowich. Manufacture of film-coated fabrics etc. 8968. Mar. 23.  
 Little, Inc. Cellulose derivative and process of preparing same. 7569. Mar. 9. (U.S., 10,420.)  
 Little, Inc. Preparation of cellulose butyrate. 7570. Mar. 9. (U.S., 26,720.)

## COMPLETE SPECIFICATIONS ACCEPTED.

9789 (1918). Mayfield. Treatment of silk and other yarns to be worked up into fabrics. (159,907.) Mar. 23.  
 22,187 (1919). Carlsson and Tball. Reducing the viscosity of solutions of nitrocellulose and its compositions. (136,141.) Mar. 23.  
 30,219 (1919). Dreyfus. Manufacture of plastic masses. (160,225.) Mar. 31.  
 1355 (1920). Naefe. Protecting woollen fabrics from moths. (160,039.) Mar. 23.  
 3342 (1920). Newsprint Reclaiming Corp. Removal of printers' ink from printed matter. (138,628.) Mar. 31.  
 16,458 (1920). Folien u. Flitterfabr. A.-G. Process for obviating the high inflammability of celluloid. (145,430.) Mar. 16.  
 18,477 (1920). Jerock and others. *See XII.*  
 19,316 (1920). Zellstoff-Fabr. Waldhof, and Schneider. Charging cellulose boilers etc. (147,417.) Mar. 31.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

## APPLICATIONS.

Aris. Employment of mordants in dye industry. 9192. Mar. 24.  
 Burgess, Ledward, and Co., and Harrison. Dyeing acetyloellulose. 8648. Mar. 21.  
 Fish, Gass, Hammond, and Jackson and Bro., Ltd. Dyeing-apparatus. 9040. Mar. 24.  
 Hatfield and Restall. Dyeing etc. processes. 7247. Mar. 7.  
 Lee. Machines for dyeing hanks etc. of yarn. 8591. Mar. 19.  
 Schofield. Sulphur bleaching of wool fabrics. 7926. Mar. 14.  
 Taylor. Machines for dyeing and padding or treating fabrics etc. 7367. Mar. 8.

## COMPLETE SPECIFICATIONS ACCEPTED.

26,565 (1919) and 17,823 (1920). Petrie and Boardman. Dyeing apparatus. (159,548.) Mar. 16.  
 26,881 (1919). Calico Printers' Assoc., Ashton, and Nelson. Printing textile fabrics. (159,552.) Mar. 16.  
 10,429 (1920). Nagelin. Apparatus for treating textiles in hank form by means of circulating liquid. (152,299.) Mar. 23.

16,409 (1920). Wioland. Dyeing and bleaching apparatus. (146,945.) Mar. 23.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

##### APPLICATIONS.

Benkő. Manufacture of iodine etc. compounds. 9203. Mar. 24. (Hungary, 1.6.20.)  
 Bloxam (Edwards). Treatment of mineral phosphate. 9171. Mar. 24.  
 Broadbridge, Edser, and Sellers. Treatment of caliche. 7533. Mar. 9.  
 Carpmæl (Bayer u. Co.). 8393. See XIII.  
 Chem. Fabr. Griesheim-Elektron, and Reitz. Rendering calcium hypochlorite stable. 9025. Mar. 23.  
 Chem. Fabr. Weissenstein. Process for distilling sulphuric acid. 8988. Mar. 23. (Austria, 21.5.20.)  
 Duets. Gold- u. Silber-Scheide-Anstalt, and Liebknecht. Generation of hydrocyanic acid. 8147. Mar. 15.  
 Evans, Parrish, Weight, and South Metropolitan Gas Co. Manufacture of ammonium sulphate. 8515. Mar. 18.  
 Goldschmidt. Manufacture of magnesium chloride. 7597. Mar. 9. (Norway, 29.3.20.)  
 Jones and Kelly. Production of alkali pentaborates direct from boron ores. 7879. Mar. 12.  
 Nitrogen Corp. Production of sodium bicarbonate. 7408. Mar. 8. (U.S., 11.3.20.)  
 Parrish and others. 7588. See II.  
 Phillipson and Thwaite. Sulphate of ammonia drier and neutraliser. 7754. Mar. 11.  
 Preston. 7237. See II.  
 Soc. l'Air Liquide. Production of sodium bicarbonate and ammonium chloride. 8133. Mar. 15. (Fr., 17.3.20.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

15,756 (1918). Soc. Indus. de Prod. Chim. See II.  
 29,293 (1919). New Jersey Zinc Co. Manufacture of zinc oxide. (137,513.) Mar. 16.  
 29,539 (1919). New Jersey Zinc Co. Production of metal oxides and other compounds of metals. (149,927.) Mar. 16.  
 29,754 (1919). Reid. Concentration of sea water. (159,592.) Mar. 16.  
 30,816 (1919). Newall. See XI.  
 30,950 (1919). Collins. See X.  
 32,763 (1919). Loring. See XI.  
 5040 (1920). Kilburn (Norske Akt. f. Elektrokemisk Ind.). Removal of iron from solutions of aluminium sulphate, nitrate, or chloride. (139,470.) Mar. 16.  
 11,814 (1920). Hauenschild. See IX.  
 16,209 (1920). Badische Anilin u. Soda Fabr. Manufacture of the hydrogen-nitrogen mixture in the synthetic production of ammonia. (145,058.) Mar. 31.  
 25,423 (1920). Zack. Separation of oxygen and nitrogen. (152,643.) Mar. 16.

#### VIII.—GLASS; CERAMICS.

##### APPLICATIONS.

Arnold and Langwell. Manufacture of glass. 7963. Mar. 14.  
 Gaved. Recovery of china clay. 7792. Mar. 11.  
 Marks (Lava Crucible Co. of Pittsburg). Production of ceramic articles. 7402. Mar. 8.  
 Stubbs. Bleaching earthy materials. 7893. Mar. 12.

##### COMPLETE SPECIFICATIONS ACCEPTED.

18,283 (1919). Marlow. Gas-fired oven or kiln for making tiles, pottery, etc. (159,522.) Mar. 16.

20,763 (1919) and 5698 (1920). Grace. Drying china clay etc. (159,525.) Mar. 16.  
 28,886 (1919). Curd. Gas-fired glass-melting furnaces. (159,559.) Mar. 16.  
 31,877 (1919). Laycock and Laycock. Annealing lehrs or kilns for glass etc. (160,282.) Mar. 31.  
 2077 (1920). Osmosis Co., Laurie, and Ormandy. Manufacture of refractory articles from china clay. (159,737.) Mar. 16.  
 12,143 (1920). Dimitri and Delaunay. Refractory and insulating product. (142,512.) Mar. 31.

#### IX.—BUILDING MATERIALS.

##### APPLICATIONS.

Brown and Earle. Mixing and application of slag cement. 9138. Mar. 24.  
 Merz and McLellan, and Weeks. Cement manufacture. 8403. Mar. 17.

##### COMPLETE SPECIFICATIONS ACCEPTED.

30,382 (1919). Harden. Acid-proof and insulating materials. (159,956.) Mar. 23.  
 11,814 (1920). Hauenschild. Burning or calcining cement materials, magnesite, etc. (159,780.) Mar. 16.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

##### APPLICATIONS.

Ampère Ges., Diegenthüler, and Rothe. Manufacture of molybdenum or its alloys. 7906. Mar. 12. (Ger., 12.3.20.)  
 Ballantine. Manufacture of ferrochrome alloys. 8334. Mar. 17.  
 Canning, and Canning and Co. Electroplating apparatus. 7806. Mar. 12.  
 Coles. Apparatus for sherardising. 7256. Mar. 7.  
 Craig, Pearson, and Durelco, Ltd. Reduction of oxides of tungsten and molybdenum. 8858. Mar. 22.  
 Elliott. Manufacture of wrought iron and steel. 7353. Mar. 8.  
 Franz. Flotation processes of dressing ores. 7451. Mar. 8.  
 Franz. Destroying froth formed in flotation processes. 7452. Mar. 8.  
 Franz. Comminuting ores etc. 7453. Mar. 8.  
 Gillespie. Manufacture of metallic powders. 8705. Mar. 21.  
 Goskar and Hitch. Compositions for case-hardening iron and steel. 9163. Mar. 24.  
 Holström and Malmberg. Determination of percentage of carbon in iron and steel. 7797. Mar. 11.  
 Liebreich. Electrolytic separation of chromium. 7425. Mar. 8. (Ger., 8.3.20.)  
 Madsen. Electro-deposition of metals. 7297. Mar. 7.  
 Pacz. Production of alloys. 8529. Mar. 18. (U.S., 18.3.20.)  
 Pacz. Producing high temperatures and reducing refractory oxides. 8530. Mar. 18. (U.S., 19.3.20.)  
 Passalacqua. Soldering aluminium. 9207. Mar. 24. (Fr., 11.6.20.)  
 Pérez. Decomposition of mercury. 7542. Mar. 9.  
 Perkins. Metallurgy of oxidised iron ores etc. 8524. Mar. 18.  
 Ramsden, and Shropshire Mines, Ltd. Treatment of ores. 8818. Mar. 22.  
 Schaufelberg. Anticarbonising paint for case-hardening mild steel and iron. 8977. Mar. 23.  
 Steen. Granulating and separating moisture from slag. 8994. Mar. 23.  
 Taylor. Cupola furnaces. 9063. Mar. 24.  
 Vautin. Treatment of iron ores. 8522. Mar. 18.

Wade (Ballantine). Manufacture of ferro-vanadium alloys. 8523. Mar. 18.  
Walter. Alloys. 9116. Mar. 24. (Ger., 24.3.20.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

5398 (1918). Estelle. Electrolytic production of iron and its alloys. (159,906.) Mar. 23.  
24,721 (1919). British Oxygen Co., Bray, and Balfour. Furnaces for subjecting ores etc. to the action of gases etc. (159,530.) Mar. 16.  
25,687 (1919). Kjellberg. Treatment of minerals or products containing iron, titanium, and vanadium. (159,532.) Mar. 16.  
26,136 (1919). Armstrong, Whitworth, and Co., McGuckin, and Smalley. Copper alloys. (159,537.) Mar. 16.  
29,216 and 31,761 (1919) and 1137 (1920). Ballantine. Production of ferrochrome alloys. (159,568.) Mar. 16.  
30,502 (1919). Nitro-Fixation Synd., and Jenkins. Manufacture of catalytic iron or catalysts containing catalytic iron. (159,960.) Mar. 23.  
30,950 (1919). Collins. Purification of tin or production of tin salts from crude tin. (159,659.) Mar. 16.  
10,756 (1920). British Thomson-Houston Co. (General Electric Co.). Tungsten alloy. (160,373.) Mar. 31.  
17,388 (1920). Krupp A.-G. Production of low-carbon ferrochromium. (145,709.) Mar. 16.  
21,155 (1920). Phillips and Arnold. Solder. (159,797.) Mar. 16.

### XI—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Canning. 7896. See X.  
Case. Photo-electric cells. 8943—4. Mar. 23.  
Coulson. Accumulators. 7781. Mar. 11.  
Darimont. Primary cells. 8256. Mar. 16.  
Elektrizitäts A.-G. vorm. Schuckert u. Co. Electrolytic cell. 7973. Mar. 14. (Ger., 14.6.20.)  
Liebreich. 7425. See X.  
Madsen. 7297. See X.  
Marks (Akt. Kfayveindustri). Heating material in electric furnaces. 9191. Mar. 24.  
Plauson. Carrying out electrochemical reactions. 2131—2. Mar. 24.  
Vogel. Electric storage cells. 8736. Mar. 21.  
Wade (Hazlett Storage Battery Co.). Manufacture of storage batteries. 9024. Mar. 23.

#### COMPLETE SPECIFICATIONS ACCEPTED.

5398 (1918). Estelle. See X.  
30,382 (1919). Harden. See IX.  
30,816 (1919). Newall. Electric furnaces for calcining magnesia etc. (160,231.) Mar. 31.  
32,763 (1919). Loring. Electrodes for oxidising nitrogen. (159,709.) Mar. 16.  
638 (1920). Allison (Narabayashi). Filling for storage batteries. (160,317.) Mar. 31.  
7664 (1920). Marks (Scovill Manuf. Co.). Electric furnaces. (160,082.) Mar. 23.  
12,143 (1920). Dimitri and Delaunay. See VIII.  
12,567 (1920). Allen. Electrodes for storage batteries. (159,782.) Mar. 16.  
17,348 (1920). Pechkranz. Electrolysers. (146,184.) Mar. 31.

### XII—FATS; OILS; WAXES.

#### APPLICATIONS.

Clifford. Soap. 8602. Mar. 19.  
Gleitz. Removal of acids from glycerides. 8908. Mar. 23.  
Heys and Macpherson. Antiseptic and insecticidal soap etc. 8040. Mar. 15.

Kayser. Recovery of glycerin from soap lyes etc. 8739. Mar. 21.

MacIlwaine. Extraction of oil by volatile solvents. 7713. Mar. 10.

Paley Engineering Co. Manufacture of soap. 8264. Mar. 16. (U.S., 1.4.20.)

Schicht. Manufacture of synthetic waxes. 8409. Mar. 17.

Soc. Anon. l'Oxydrique Française. Removal of catalysts from oils and fatty bodies treated therewith. 8622. Mar. 19. (Fr., 20.3.20.)

Tseng. Manufacture of transparent soap. 8428. Mar. 18.

#### COMPLETE SPECIFICATIONS ACCEPTED.

29,705 (1919). Bolton and Lush. Neutralisation of fatty acids in fats and oils. (159,587.) Mar. 16.

1113 (1920). Niessen. Apparatus for treating mixtures of fat and glue water derived from boiling and drying organic substances. (137,842.) Mar. 23.

18,477 (1920). Jerock, and Reichsausschuss für pflanzliche u. tierische Öle u. Fette. Manufacture of fat from spent sulphite cellulose lyes. (146,430.) Mar. 31.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

Boularan. Paint etc. 8513. Mar. 18.  
Carpmael (Bayer u. Co.). Manufacture of lithopones and barium hydroxide. 8393. Mar. 17.  
Günther. Oil and varnish colours soluble in water. 7236. Mar. 7.  
Locke. Coating-materials. 9197. Mar. 24. (U.S., 24.3.20.)  
Parker. Water paint. 9057. Mar. 24.  
Waele. Inks, pigmenting etc. compositions. 8608. Mar. 19.

#### COMPLETE SPECIFICATIONS ACCEPTED.

26,426 (1919). Ivinson and Roberts. Composition impervious to oils, spirits, water, etc. (159,542.) Mar. 16.

26,427 (1919). Ivinson and Roberts. Anticorrosive paint or composition. (159,543.) Mar. 16.

31,392 (1919). Vickers, Ltd., Ioco Rubber and Waterproofing Co., and Nuttall. Condensation of phenolic bodies with aldehydic compounds. (160,258.) Mar. 31.

1814 (1920). Dunham. Production of dry, water-soluble products from karaya gum. (160,045.) Mar. 23.

7489 (1920). Pooley and Stevens. Extraction of gum from grass trees. (160,080.) Mar. 23.

18,079 (1920). Dahl. Apparatus for making white lead. (160,395.) Mar. 31.

28,124 (1920). Fitzgerald. Lithographic inks. (159,809.) Mar. 16.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### APPLICATIONS.

Bilbrough. Manufacture of rubber sponges, paints, solutions, and adhesives, etc. 8355. Mar. 17.

Bouvery and Conort. Recovery of rubber. 8533. Mar. 18. (Fr., 26.3.20.)

Davidson. Preparation of raw rubber. 7563. Mar. 9.

Wheatley. Heat vulcanisation of rubber. 8313. Mar. 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

29,930 (1919) and 1691 (1920). Davidson. Manufacture of rubber. (159,602.) Mar. 16.

30,870 (1919). Willard. Devulcanising vulcanised rubber. (159,987.) Mar. 23.

#### XV.—LEATHER; BONE; HORN; GLUE.

##### APPLICATIONS.

Dobson. Composition for dyeing or colouring leather etc. articles. 9169. Mar. 24.  
Jones. Insoluble size. 7716. Mar. 10.  
Strain. Manufacture of sheet gelatin. 8071. Mar. 15.

##### COMPLETE SPECIFICATIONS ACCEPTED.

23,347 (1919). Fairrie. Drying leather etc. by means of gases. (160,197.) Mar. 31.  
30,516 (1919). Gilardini. Apparatus for rapid tanning of hides and skins. (160,422.) Mar. 31.  
1113 (1920). Niessen. See XII.

#### XVI.—SOILS; FERTILISERS.

##### APPLICATION.

Roberts. Manufacture of fertilisers. 8732. Mar. 21.

##### COMPLETE SPECIFICATION ACCEPTED.

6539 (1920). Chemical Construction Co. Apparatus for making superphosphate of lime and similar compounds. (139,803.) Mar. 31.

#### XVII.—SUGARS; STARCHES; GUMS.

##### APPLICATIONS.

Acree. Converting wood into sugar etc. 8492. Mar. 18. (U.S., 25.3.20.)  
Bloxam (Kantorowicz). Manufacture of starch paste. 8989. Mar. 23.  
Bloxam (Kantorowicz). Manufacture of an adhesive from potato starch. 8990. Mar. 23.  
Manss. Treatment of sugar juice. 7456. Mar. 8.

##### COMPLETE SPECIFICATION ACCEPTED.

30,541 (1919). Grant. Decolorising and purifying sugar. (159,640.) Mar. 16.

#### XVIII.—FERMENTATION INDUSTRIES.

##### APPLICATIONS.

Briscoe. Brewing beer etc. 7945. Mar. 14.  
Seligman. Vessels for brewing beer etc. 8396. Mar. 17.

#### XIX.—FOODS; WATER PURIFICATION; SANITATION.

##### APPLICATIONS.

Barnes and Hyatt. Purification and clarification of water or treatment of sewage. 7813. Mar. 11.  
Daw. Treatment of factory waste etc. 7305. Mar. 7.  
Dorr Co. Treatment of sewage. 8275. Mar. 16. (U.S., 31.7.20.)  
Dorr Co. Apparatus for treating sewage. 9015. Mar. 23. (U.S., 14.10.20.)  
Heys and Macpherson. 8040. See XII.  
Rothe. Production of a food containing aerobic bacteria. 8800. Mar. 22.  
Shields. Foodstuff. 8565. Mar. 19.  
Soc. Anon. de Prod. Chim. Etabl. Malétra. Manufacture of a fungicide and insecticide. 7959. Mar. 14. (Fr., 13.3.20.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

27,401 (1919). Barbet et Fils et Cie. Concentration of fruit juice in the manufacture of grape honey and fruit syrups. (135,175.) Mar. 23.

30,979 (1919). Townsend. Manufacture of milk food products. (160,224.) Mar. 31.  
8276 (1920). Lombaers. See XX.

#### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

##### APPLICATIONS.

Acree. Converting wood into mucic acid etc. 8493. Mar. 18. (U.S., 25.3.20.)  
Barrett Co. Manufacture of formaldehyde. 8302. Mar. 16. (U.S., 26.5.20.)  
Bloxam (Chem. Fabr. Griesheim-Elektron). Manufacture of liquid esters of phosphoric acid. 8825. Mar. 22.  
Imray (Soc. Chem. Ind. Basle). 8516. See IV.  
Mond (Metallbank u. Metallurgische Ges.). Evaporating, concentrating, and drying urea solutions. 8343. Mar. 17.  
Thron, and Verein. Chininfabr. Zimmer u. Co. Manufacture of O-alkyl derivatives of hydrocupreine. 8298. Mar. 16.  
Wallis. Extraction of thymol. 8485. Mar. 18.

##### COMPLETE SPECIFICATIONS ACCEPTED.

3384 (1917). Elektro-Osmose A.-G. Purifying and enriching immunising serum. (104,688.) Mar. 16.  
10,050 (1919). Marks (New Jersey Testing Laboratories). Production of reactive acid liquor alcohols, esters, etc. from olefine hydrocarbons. (160,185.) Mar. 31.  
30,706 (1919). Fabr. Prod. Chim. Thann et Mulhouse. Manufacture of borneol. (144,604.) Mar. 31.  
8276 (1920). Lombaers. Extraction of caffeine from coffee beans. (144,998.) Mar. 23.  
14,516 (1920). Chem. Fabr. Griesheim-Elektron. Manufacture of acetaldehyde from acetylene. (143,891.) Mar. 31.

#### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

##### APPLICATIONS.

Drysdale, Powell, and Wood. Sensitised photographic films. 8107. Mar. 15.  
Hamburger. Photographic processes. 8969. Mar. 23.  
Kelley. Treating and dyeing photographic images. 7788. Mar. 11. (U.S., 25.4.18.)  
Lage. Producing photographic plates for indirect tri-colour photography. 8590. Mar. 19.  
Martinez. Direct-colour photography and/or cinematography. 7517. Mar. 9.  
Martinez. Colour cinematography by interference of rays. 7522. Mar. 9.  
Obergaßner. Producing opaque photographs in natural colours. 8695. Mar. 21.

#### XXII.—EXPLOSIVES; MATCHES.

##### COMPLETE SPECIFICATIONS ACCEPTED.

18,183 (1920). Herz. Explosives. (145,791.) Mar. 23.  
18,195 (1920). Duhrisay. Manufacture of damp-proof matches. (145,798.) Mar. 23.

#### XXIII.—ANALYSIS.

##### APPLICATIONS.

Cammell, Laird, and Co., Carter, and Ramsay. Pyrometers. 7511. Mar. 9.  
Holström and Malmberg. 7797. See X.  
Taylor. Refractometers. 8322. Mar. 17.

##### COMPLETE SPECIFICATION ACCEPTED.

13,884 (1916). Aktiebol. Ingeniörsfirma F. Egnell. Gas-analysing apparatus. (106,265.) Mar. 16.

## I.—GENERAL; PLANT; MACHINERY.

## PATENTS.

*Heat of liquids; Apparatus for utilizing the — for heating other liquids.* E. Wirth-Frey. E.P. 125,368, 21.3.19. Conv., 12.4.18.

VAPOUR of a liquid contained in one vessel is passed into a liquid contained in another vessel by means of a suction and compression pump between the two vessels, or by means of a vacuum pump drawing from the top of the second vessel. Several vessels may be interconnected, so that heat may be transferred from any one to any other, and air may be bubbled through both the liquids to assist the process.—B. M. V.

*Drying material containing liquid; Process and apparatus for —.* G. Schjelderup. E.P. 139,478, 23.2.20. Conv., 24.2.19.

A DRYER, specially suitable for ore flotation concentrates, is formed of a series of superposed metallic hearths somewhat similar to a mechanical roaster, the material being rabbled from circumference to centre and *vice versa* on alternate hearths. The vapour arising from the material being dried is drawn off, compressed, and returned to the interiors of the hearths, where it condenses and gives up its heat to the material; the vapours pass upwards through all the hearths in succession, counter-current to the material being dried, and to afford the initial heat to prevent condensation in the drying chambers the material is preheated, part of which heat may be derived from the heat remaining in the condensed liquid. The admission of wet and withdrawal of dry material may be effected through airtight doors so that there is no loss of dust or vapour.—B. M. V.

*Drying-stoves and the like; Gas-fired —.* W. Muirhead. E.P. 159,393, 13.1.20.

DRYING chamber is connected with a pair of longitudinal regenerators below by a series of openings along each side at the bottom. Gas is burned in one of the regenerators, and the hot gas passes transversely across the drying chamber to the other regenerator, which is thus heated. The functions of the two regenerators may be reversed by dampers controlling the air and gas passages.—W. F. F.

*Drying plant.* P. Barducci. U.S.P. 1,368,537, 15.2.21. Appl., 19.1.20.

MATERIAL is placed in a drying room through which carriage travels horizontally. The carriage comprises a casing with a horizontal top, in which are openings arranged in lines transverse to the path of the casing, and an internal propelling device which causes a circulation of air through the casing and around a horizontal axis in the room.

—W. F. F.

*Heater for drying apparatus.* O. Pfeiffer. G.P. 329,828, 8.2.19.

AIR to be heated and the heating gases flow through a series of chambers constructed of parallel plates, the opposite sides of alternate chambers being open, so that the paths of the air and of the heating gases cross one another at right angles.

—L. A. C.

*Drying apparatus; Rotary — [for drying yeast or milk or other material liable to froth].* L. Eberts. G.P. 330,129, 1.5.18.

A feed tube, provided with a slit through which the material is supplied to the drying surface, is connected with an overflow tube which returns excess material to the reservoir.—L. A. C.

*Separation of a solvent from a material with simultaneous drying; Process and apparatus for —.* Trocknungs-Anlagen G.m.b.H. G.P. 330,449, 19.9.19.

A BLAST of cold air is injected into the hot solvent vapour, which is thereby condensed and falls into a receiver, while the warm air is employed for drying the material.—L. A. C.

*Furnace for the continuous drying or ignition of substances without exposure to a direct fire.* Farbenfabr. vorm. F. Bayer und Co. G.P. 331,784, 18.11.17.

THE substances to be dried are enclosed in drums which, by means of rings running on rails, are made to move down the inclined hearth of a furnace. The revolution of the drums serves to keep the contents thoroughly mixed.—A. R. P.

*Dryers; Stirring apparatus for revolving tube —.* Maschinenfabr. Buckau A.-G. zu Magdeburg. G.P. 332,050, 27.1.18.

A FREELY-MOVING cylindrical body of smaller cross-section than the drying tube is placed inside it, so that during the revolution of the tube it rolls round the walls and not only shakes the contents up, but stirs them about. Means are provided to prevent the roller from falling out of the tube.—A. R. P.

*Separating solid substances from gases and vapours, more especially blast furnace gases; Apparatus for —.* Deutsche Maschinenfabrik A.-G. E.P. 148,802, 10.7.20. Conv., 2.12.18.

THE filtering surfaces are built up of a number of units, which can be withdrawn individually or in groups, a number of rings of such units being assembled one above the other, so as to form a central outlet conduit for clean gas. The sectional plan view may be similar to a cog wheel with very deep teeth.—B. M. V.

*Electrical treatment of gases; Apparatus for —.* A. Mond. From International Precipitation Co. E.P. 158,982, 13.11.19.

AN electrical precipitator for removing suspended particles from gases comprises vertical collecting electrodes consisting of tubes or plates of relatively large cross-section and discharge electrodes consisting of smooth bars, rods, or twisted square rods of metal, of relatively small cross-section, arranged about a common axis within or between the collecting electrodes. The discharge electrodes are supported at their upper or lower ends only. Matter deposited upon the collecting and discharge electrodes is dislodged therefrom by blows delivered by hammers to plates mounted on and between adjacent collecting electrodes and to the supports of the discharge electrodes. Means are provided whereby such blows may be delivered without interrupting the electric current.—J. S. G. T.

*Discharge electrode for high-tension current used in the purification of gases or vapours.* H. Zschocke. G.P. 331,381, 21.10.19.

METALLIC bands in the form of rings or helices are wound upon the two limbs of a U-shaped, perforated hollow body, so that there is a narrow gap between adjacent windings, through which either purified or unpurified gas may be passed. When the gas is not heavily charged with dust particles the unpurified gas is fed to the interior of the electrode. In other cases, as, for example, when the gas is heavily charged with dust particles which might choke up the narrow slots, a small current of purified gas is fed to the electrode, while the crude gas is fed longitudinally between the discharge and precipitating electrodes.—J. S. G. T.

*Electrode for use in the [electrical] deposition of dust.* H. Thein. G.P. 331,590, 20.8.19.

THE electrode is constituted of a flexible hollow body such as metallic tubing.—J. S. G. T.

*Electrical purification of gases; Process for the removal of dust deposited in the —.* H. Zschöcke. G.P. 332,110, 5.11.19. Addn. to 329,062 (J., 1921, 205 A).

THE deposition tubes and the axes thereof are rotated. The tubes may also be moved simultaneously in an axial direction. The particular motion imparted to the tubes is determined according to the compactness of the dust deposited.

—J. S. G. T.

*Gas filter.* W. M. Bovard, Assr. to N. D. Baker. U.S.P. 1,368,540, 15.2.21. Appl., 14.1.19. Renewed 24.12.20.

A gas filter is composed of a porous mat of animal and vegetable fibres, mixed with porous mineral matter.—W. F. F.

*Filter chamber containing metallic filling for purifying gases.* E. Fernholz. G.P. 330,356, 7.3.20.

THE filling bodies are connected together, e.g., by soldering, to avoid the necessity for supporting the bodies by perforated plates to prevent uneven packing and formation of gas pockets.—L. A. C.

*Gas filters containing granular or fibrous material; Method of attaining uniform filtrations with —.* Tellus A.-G. für Bergbau und Hüttenindustrie. G.P. 330,450, 7.11.19.

THE filter is packed so that the permeability of the filtering material decreases in the direction of the current of gas.—L. A. C.

*Precipitation of solid or liquid impurities from streams of gas or vapour; Apparatus for —.* G. Klingenberg. G.P. 332,164, 5.9.19.

A NUMBER of plates are arranged transversely to the gas stream, and the spaces between the plates are divided by partitions at right angles to the plates. Both plates and partitions are provided with openings, which are so disposed that the gas in passing between successive plates has to flow through the openings in the partitions. Deposition of solid and liquid particles is effected owing to the change of direction of the stream, and by the retardation occurring when the cross-sectional area increases. Solid particles may be collected and removed by a stream of fluid flowing at right angles to the main gas stream.—J. S. G. T.

*Gas scrubber.* A.-G. der Dillinger Hüttenwerke, and R. Kunz. G.P. 326,964, 22.11.18.

THE scrubber is filled with alternate layers of distributing cones and collecting funnels, and the upper surfaces of the cones and funnels are provided with radial grooves to equalise the distribution of the water and thus increase the efficiency of the scrubbing action.—L. A. C.

*Separating aqueous and other vapours from liquids and solids, and preparing dilute sulphuric acid; Process of —.* O. Maass. E.P. 159,054, 3.12.19.

MATERIAL to be treated is contained in a vessel which is evacuated as completely as possible by oscillation of an absorbing or desiccating agent, e.g., concentrated sulphuric acid, in a suitable pumping device connected with the vessel. During such oscillation the desiccating agent passes over a distributing surface, whereby large superficial contact is maintained between it and the vapour derived from the material under treatment. If necessary, heat may be applied to the material. The process may be applied to the concentration

of weak solutions of hydrogen peroxide, the preparation of milk powder, etc.—J. S. G. T.

*Crushing mills of the roller and ring type.* C. M. Conder and G. T. Vivian. E.P. 159,244, 10.10.19.

A PAIR of rollers and a ring inside which they run are all supported on parallel horizontal shafts, one of the roller shafts and the ring shaft having sliding bearings, so that each roller may be pressed equally on the ring by means of springs between the two bearing boxes. The pressure can be adjusted by a nut turning upon a screwed pin inside one of the bearing boxes, the pressure being taken off the threads after adjustment by means of shims. The top half of the casing of the machine is in two parts, each part being hinged to the lower part of the machine.—B. M. V.

*Pulverising machine.* O. A. Kreutzberg. U.S.P. 1,368,636, 15.2.21. Appl., 8.11.19. Renewed 8.7.20.

TWO cones of wide angle are mounted on separate parallel shafts so that their conical surfaces are in line contact. One cone is provided with a segment of its conical surface slightly raised above the remainder, and the other cone is movable axially on its shaft against a spring to allow the passage of the raised portion past the line of contact.

—W. F. F.

*Mill; Ball-grinding —.* P. T. Lindhard, Assr. to F. L. Smidth & Co. U.S.P. 1,368,739, 15.2.21. Appl., 15.5.19.

MATERIAL is fed through a horizontal axial passage into a horizontal grinding drum, and passes through a perforated transverse plate at the other end on to a cylindrical screen. The finer material passes through the screen to the discharge, and the coarser material is collected by scoops and conveyed to an axial conduit passing through the discharge conduit, back to the grinding drum.—W. F. F.

*Pulverising or comminuting apparatus.* C. E. Tetley. U.S.P. 1,369,061, 22.2.21. Appl., 10.7.19.

A GRINDING apparatus comprises a series of short drums placed concentrically end to end and communicating with one another. The inner surface of each drum is curved to a barrel shape, and the successive drums are of decreasing size from the inlet to the outlet end. The drums are rotated on their axes, and each is provided with a loose ball.

—W. F. F.

*Ball or tube mills; Discharge spout for —.* N. L. Hall. U.S.P. 1,369,653, 22.2.21. Appl., 2.8.20.

THE horizontal hollow discharge trunnion of a rotating ball or tube mill is provided with a number of short internal vanes with helical surfaces. The heavier material is intercepted and returned to the mill by the vanes, and the lighter material is discharged.—W. F. F.

*Furnaces; Recuperative —.* W. S. Rockwell, Assr. to W. S. Rockwell Co. U.S.P. 1,358,390, 9.11.20. Appl., 4.11.19.

A HEATING chamber is provided with burners arranged along the side walls and delivering gas into mixing chambers in the walls. Each air supply conduit passes upwards through the side wall and enters the mixing chamber at the bottom, while the delivery port into the furnace is constricted sufficiently to draw in the air through the supply passage. The combustion products pass from the heating chamber through openings at the bottom into a chamber below, which extends the full length and width of the floor, and the gases then pass in a tortuous path through a pair of regenerators below, which preheat the air supply.—W. F. F.

*Furnace; Refractory* — W. J. Hill. U.S.P. 1,363,787, 23.12.20. Appl., 5.3.20.

THE lower part of the heating chamber is filled with a refractory radiating element consisting of layers of bricks arranged alternately in longitudinal and transverse rows with intervening spaces. A central longitudinal outlet flue runs along the bottom of the structure and is provided with side openings leading to side flues. A flame is projected horizontally into the upper part of the furnace from a burner at one end, and is deflected downwards through the radiating element by a horizontal plate surmounted by a vertical longitudinal plate, which lies along the top of the structure. The horizontal plate is arranged centrally but is of less width than the furnace.

—W. F. F.

*Reverberatory furnace; Process for heating solid material in a* — Magnesit-Ind. A.-G. G.P. 329,325, 20.12.18.

THE solid hearth is replaced by a cooling shaft into which the material falls, the portion of the material immediately under the influence of the flame being supported by the material cooling in the shaft.

—L. A. C.

*Air-separator*. T. J. Sturtevant, Assr. to Sturtevant Mill Co. U.S.P. (A) 1,367,635, (B) 1,367,636, and (C) 1,367,637, 8.2.21. Appl., (A) 24.1.20, (B) (C) 16.2.20.

(A) MATERIAL is fed into an air current by another air current which is caused to whirl and so project the material transversely into the separating current. (B) The separating air current moves upwards in a chamber, and the material is fed into the chamber by another current moving upwards. The feeding current is received by a rotating device which whirls it horizontally into the separating current. (C) Air is circulated upwards through a separating chamber and then back through a by-pass to the bottom of the chamber, while material to be graded is introduced into the by-pass. The mixture is whirled by a fan into an outer collecting chamber and the material is removed before the air passes back to the by-pass.—W. F. F.

*Mixing granular substances; Apparatus for* — O. Krause. U.S.P. 1,369,248, 22.2.21. Appl., 23.11.20.

THE substances to be mixed are supplied through separate discharge shoots, along which the substances slide and from which they fall freely through the air in a parabolic path. The shoots are so inclined that the substances all meet at a common point in their fall and follow each other throughout the remainder of the fall as a uniform mass to a place of collection.—D. W.

*Still*. A. C. Jewell. U.S.P. 1,369,438, 22.2.21. Appl., 23.10.17.

THE vapour generated in a still passes upwards through a vertical column which opens at the top into an annular chamber surrounding the column. The wall of the column, which forms the inner wall of the annular chamber, is made double, and a current of air is passed upwards through the jacket thus formed which opens at the top into the annular chamber. The annular chamber is surrounded by a water jacket and acts as a condenser.—W. F. F.

*Salts or other granular or slimy material; Apparatus for washing* — E. Waskowsky. G.P. 328,752, 22.8.17, and 330,255, 14.8.18.

THE apparatus consists of two washers worked on the counter-current principle and provided with a conveyor the blades of which may, if necessary, be perforated and provided with outer rims. The con-

veyor may be in the shape of a drum with a solid or perforated casing, and the washing apparatus is then provided with a sieve, from underneath which the washed material is removed.—A. R. P.

*Washing granular filter material by means of compressed air and water; Process for* — K. Morawe. G.P. 323,943, 23.1.19. Addn. to 311,593 (J., 1919, 522 A).

ALTERNATE lateral motion and rearrangement of the filter material is effected by the operation of a siphon, whereby, alternately with the addition of washing water, a corresponding mass of sludge is raised above the filter bed. The lower limb of the siphon passes into the filter bed and a plate arranged at the entrance to the siphon prevents the filter material being carried over.—J. S. G. T.

*Filter press and frame, in which the filter cloths are separated by lattice work*. K. u. Th. Möller G.m.b.H. G.P. 332,163, 28.4.20.

THE lattice employed is designed so that its profile shows a double flange. The filter surface may then be increased, as the number of supports of the filter cloth upon the double flanges may be varied according to the nature of the liquid to be filtered, and the pressure employed can be altered by variation of the depth of profile and of the distance between the lattices. Corrugated cross-pieces may be used to form the lattices, which may be installed at a distance from one another, leaving an unrestricted channel between.—J. S. G. T.

*Solutions; Evaporating atomised* — Evaporating and effecting chemical reactions in atomised solutions. G. A. Krause und Co., A.-G. G.P. (A) 329,357 and (B) 329,358, 30.1.17. Addns. to 297,338.

(A) A HORIZONTAL or inclined stream of the atomised liquid is injected into a vertical current of warm, dry gas. (B) The atomised liquid is injected in a nearly horizontal disc-shaped stream into a descending current of warm, dry gas, and the dried product is thus carried directly down to the bottom of the drying chamber.—L. A. C.

*Evaporation of solutions of salts etc.; Rotating drum for the* — G. Sauerhrey Maschinenfabrik A.-G. G.P. 330,357, 10.10.18.

THE drum is provided with lifting scoops extending the length of the drum and so arranged that the liquid circulating in the drum meets a number of transverse surfaces between the scoops at such an inclination that transport of the deposited salt along the length of the drum is effected. A central conveyor for removing the salt is thus rendered unnecessary.—J. S. G. T.

*Evaporator*. B. Graemiger. G.P. 331,792, 6.9.19.

AN evaporator with vertical heating tubes is provided with a double-walled fall tube, the outer casing of which is closed at the top where the tube for admitting fresh supplies of liquid is connected, and open at least for the greater part of its ring-like cross-section towards the container from which the series of heating tubes is fed. The fresh liquid which flows down the outer casing prevents the evaporated liquid in the inner tube from receiving so much heat that it is evaporated still further with evolution of steam bubbles and consequent hindering of its sinking.—A. R. P.

*Liquefaction of gases; Process for the* — and for producing low temperatures by releasing gases under pressure. B. Haak. G.P. 329,361, 15.11.14.

THE necessary high pressure is attained by the electrolytic generation of the gas under pressure; e.g., liquid oxygen and hydrogen are prepared by



the fractional condensation of the mixture of gases obtained by the electrolysis of water.—L. A. C.

*Lining for protecting apparatus and vessels employed in chemical technology against chemical action.* F. Schüler. G.P. 329,659, 28.11.19. Addn. to 318,083 (J., 1920, 356 A).

A SINGLE layer of stoneware protected by a coating of silicate or hy glass sheets is used. Thin strips of glass are laid under the joints of the glass sheets, or folds engaging with one another may be provided in the sheets. The stoneware covered with silicate should be provided with expansion joints and these filled with acid-resisting material.

—J. S. G. T.

*Vessels having worm coils within the walls; Manufacture of —.* Maschinenfabr. Esslingen. G.P. 330,018, 12.5.18.

THE walls of the vessels are made of ferrosilicon containing 12–18% Si which is cast around an iron worm tube (cf. G.P. 306,001; J., 1918, 628 A).

—L. A. C.

*Pressing, leaching, distilling, extracting, rinsing, separating, and dissolving materials and effecting chemical reactions; Means for —.* O. Endriss. G.P. 330,225, 26.1.18.

TWO bodies of the material are compressed together so that any liquids or gases formed or expressed can flow away without employing a sieve or filter cloth, or a single body of the material is compressed against a solid wall or walls.—L. A. C.

*Filling material for reaction chambers.* J. Thede. G.P. 330,226, 25.3.20.

THOROUGH admixture of the gases in a reaction chamber, e.g., in the manufacture of sulphuric acid by the lead chamber process, is attained by providing the filling material with slanting channels of varying depth whereby variations in velocity and vortex motions are imparted to the gases flowing through the chamber.—L. A. C.

*Drying solutions; Process for — by removal of part of the solute by addition of a solid substance.* E. Harsányi and K. Medgyes. G.P. 331,142, 8.10.19. Conv., 28.11.17.

A SUFFICIENT quantity of the dried solid substance from a previous operation is added to a solution of the same substance to give on standing a homogeneous crumbly mass which may be readily dried by known methods. For example, 100 g. of gelatin, dried at 105° C. and crushed to 1 mm. size, is mixed while still warm with 55 g. of a 5% gelatin solution at 70° C.; after standing for 15–25 mins. the solution sets to a mass which readily crumbles to a powder, containing 16% of moisture. 30 g. of this product is removed and the remainder dried at 105° C. and used again in the process.—A. R. P.

*Leaching vegetable, animal, or mineral substances; Process and apparatus for —.* Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 145,045, 14.6.19. Conv., 9.1.15.

SEE G.P. 294,667 of 1915; J., 1917, 377.

*Pulverising or fine grinding; Process and apparatus for —.* F. Seymour. E.P. 159,925, 7.11.19.

SEE U.S.P. 1,363,361 of 1920; J., 1921, 135 A.

*Refrigeration; Method of —. Refrigerating apparatus.* B. Thoens. U.S.P. 1,369,365-6, 22.2.21. Appl., 28.5 and 22.12.19.

SEE E.P. 152,898 of 1919; J., 1920, 809 A.

*Recuperators for use in connexion with furnaces.* H. Hecker, and Bender und Främhs Ges. E.P. 133,046, 24.9.19. Conv., 10.9.18. Addn. to 133,045.

*Furnaces; [Silencing and heat-guarding casing for] oil-fired —.* Manlove, Alliott and Co., and F. L. Mason. E.P. 159,391, 8.1.20.

*Carboys; Packing-cases for —.* S. Percival. From Illinois Glass Co. E.P. 159,394, 16.1.20.

*Crushers.* A. Mond. From Traylor Engineering and Manufacturing Co. E.P. 159,950, 1.12.19.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Karbozit.* H. R. Trenkler. Feuerungstechn., 1921, 9, 93–95.

KARBOZIT is produced from lignite, wood, or peat by heating the raw material to a temperature not exceeding 450° C. By this means all the original water, together with a large amount of carbon dioxide, is evolved, thus concentrating the combustible matter in less mass. The material is dried to a moisture content of 10–15% by means of a mixture of air and hot waste gases at 75° C. in drying hunkers arranged over the ovens. Dry coal is sifted, the fines being gasified in producers and the large coal passed into an annular chamber, the lower part of which forms a cooler. Heat is applied by passing hot gases through the charge, a temperature of 250°–300° C. being maintained. Any tar produced is drained off below and allowed to mix with the "Karbozit." From a coal of composition, C 27.37%, H 2.00%, O and N 10.78%, S 0.54%, ash 3.23%, moisture 56.08%, and calorific value 2110 cal. per g. there was produced a fuel containing C 69.31%, H 4.12%, O and N 18.90%, S 0.91%, ash 5.32%, moisture 6.44%, and calorific value 6290 cal. per g. The great drawback to the process is that fines cannot be utilised.—A. G.

*Petroleum; Formation of — from fish oils. Origin of Japanese petroleum.* K. Kohayashi. Kōgyō-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, 24, 1–26.

By distilling a mixture of herring oil (4000 c.c., sp. gr. 0.9214, saponif. value 187.7) and finely powdered Japanese acid clay (4000 g.) covered with the same clay (4000 g.) in an iron retort at ordinary pressure, the author obtained a crude oil (2000 c.c.) and water (1800 c.c.). Among the products were acrolein, carbon dioxide, soluble fatty acids, such as acetic acid, other fatty acids, naphthenic acids, and hydrocarbons. The oil distillate (sp. gr. 0.8160 at 15°/4° C., saponif. value 15.1, percentage absorbed by concentrated sulphuric acid 14.5) had a greenish fluorescence and a petroleum-like odour and closely resembled natural crude petroleum. The hydrocarbons in the oil were mainly of the naphthene series, together with a considerable quantity of olefines. The formation of the hydrocarbons is ascribed to the action of the Japanese acid clay  $\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot x\text{H}_2\text{O}$  (J., 1920, 655 A). The oil is hydrolysed to fatty acids and glycerol by the action of water in the clay, and the fatty acids are decomposed into hydrocarbons partly with formation of acetic and naphthenic acids and mainly by the elimination of carbon dioxide. The hydrocarbons thus formed are "cracked" to lighter ones by the heated surface of the upper layer of clay. There are very close geological relations between the oil fields and the outcrops of Japanese acid clay, and Japanese petroleum is accompanied by salt water. It is suggested that Japanese petroleum and oil gas

are formed from marine animals. Fish oils, produced from fishes by putrefaction etc., are decomposed to petroleum and oil gas, according to a process similar to that described above. The naphthene hydrocarbons are probably derived from unsaturated fatty acids, and hydrocarbons of the  $C_{25}H_{52}$ -s,  $C_{25}H_{52}$ -e, etc. series from more highly unsaturated acids.—K. K.

## PATENTS.

*Coal; Apparatus for washing* — F. Courtoy. E.P. 154,167, 24.2.20. Conv., 14.11.19.

THE coal and water are fed in at the upper part of a hopper with an inclined bottom, provided with an adjustable opening for the issue of coal near the bottom and at the top with an outlet for the ash-containing slimes, carried along by the current of water; in front of the upper outlet is a vertical wall against which the scum of the slimes with smaller ash content collects. The adjustment of the coal outlet is effected automatically by means of a float guided so that when the quantity of coal fed in by the water increases the float rises, thus giving a wider passage towards the outlet and *vice versa*. The upper and lower currents can be adjusted by means of flap-valves placed between the float and the outlet.—A. G.

*Coal; Treatment of materials containing* — F. Edser, H. L. Sulman, and F. B. Jones. E.P. 159,285, 20.11.19.

THE powdered material, suspended in liquid, is subjected to agitation and/or aeration to disseminate minute air bubbles throughout the liquid in such a way that the coal particles are caught in the air-water surface of the bubbles and floated in the form of a froth and are thus separated from the gangue. The liquid consists of water containing in solution a modifying or deflocculating agent, *e.g.*, sodium silicate, silicic acid sol, sodium carbonate, caustic soda, or other suitable alkali or mixtures of these. When it is desired to separate the coal entirely, cresol or other frothing agent is added to the solution. An insoluble agent, such as paraffin oil, may be added to stabilise the froth, whereby carbonaceous shale may be floated along with the coal. Common salt may also be used in the liquid, and for the promotion of aeration, a vegetable infusion may be added, such as an infusion of saponin, cinchona, or quassia.—A. G.

*Briquetting apparatus*. C. W. G. Clewlow. E.P. 158,686, 10.11.19.

IN an apparatus for converting pulverised coke breeze or anthracite dust into briquettes for fuel, molten pitch is added to the solid material in a hopper provided with revolving paddles. The delivery nozzle for the pitch is connected with the stirring apparatus, so that the jet follows the paddles. The mixture is transferred to another vessel, where it is subjected to the action of steam and hot gas, which may be supplied through co-axial nozzles.—W. F. F.

*Pulverised fuel; Manufacture of mixtures of — with tar or other liquid fuel*. A. McD. Duckham. E.P. 159,089, 24.1.20.

TO obviate the necessity of drying the coal before grinding, and to overcome the danger caused by fine coal dust in suspension in grinding plants, the coal is ground in the presence of the liquid fuel.—A. G.

*Fuel; Process for drying wet* — C. Christians. G.P. 330,924, 17.5.13.

THE hot gaseous products of combustion of fuel are passed over the wet fuel in such a manner that the moisture is evaporated from that part of the wet

fuel that is nearest the inlet tube for the gases and condenses in that part which is more remote, from which it is removed in the liquid state by suitable outlet pipes.—A. R. P.

*Fuel; Process of oxidising* — T. J. Stephens, Assr. to Stephens Engineering Co. U.S.P. 1,369,200, 22.2.21. Appl., 26.12.18.

GROUND fuel is mixed with an oxidising medium, and the mixture is passed downwards into one side of a zone maintained at a temperature which promotes ignition, whereby the finer particles in suspension are burnt, whilst the larger particles are deposited to form a non-coherent bed. The particles which compose the portions of this bed opposite to the side at which downward projection occurs are agitated by a projected stream of additional oxidising medium.—A. G.

*Coke or carbon; Apparatus for separation of — from slag or the like*. Soc. "Le Coke Industriel." E.P. 149,654, 10.6.20. Conv., 13.8.19. Addn. to 120,932 (J., 1919, 672 A).

THE top plates of the separating compartments are in two parts and have a double joint, which allows them to be inclined and, consequently, the widths of the upper openings of the compartments to be varied at will, with a view to regulating the speed of the ascending currents of water.—A. G.

*Coke ovens*. La Soc. de Fours à Coke et d'Entreprises Industrielles. E.P. 130,974, 22.7.19. Conv., 9.8.18.

A COKE oven has reversible regenerators running longitudinally below the ovens, and vertical flues which are connected with regenerator compartments on both sides of the heating wall. Each regenerator is divided into four compartments corresponding to four sets of flues. The oven may be heated by rich gas, in which case the gas is supplied cold, the air for combustion is heated by passing through two regenerator compartments, and the combustion products pass through the other two compartments. When poor gas is used the gas is heated in two compartments of a set below one oven and the air in the corresponding compartments of the set below the adjacent oven.—W. F. F.

*Coking retort-oven*. H. Koppers, Assr. to The Koppers Development Corp. U.S.P. 1,369,673, 22.2.21. Appl., 10.9.20.

THE distillate is withdrawn from the coking charge in one direction until the distillation of the mass has been almost completed, and is then withdrawn in the opposite direction during the continuance of the coking operation. By such reversal in flow of the distillate through the charge the hotter regions of the charge are cooled and over-coking thereof is retarded. The cooler portions of the charge are also completely coked.—A. G.

*Gas producers*. C. B. Tully. E.P. 159,409, 19.2.20.

IN gas plants where steam is supplied intermittently to the producer, and where the gas is led off past a gas valve of the lift type immersed in liquid within a box, a lever for moving the gas valve is interconnected with the steam valve used for admitting steam to the producer, and hand-operated means are provided so that the gas and steam valves can be opened or closed together.—A. G.

*Fuel; Manufacture of — from crude petroleum oil or residues thereof by distillation*. H. Wade. From Standard Oil Co. E.P. 158,918, 21.7.19.

THE oil is distilled continuously by passage through a series of stills which are fired externally and heated internally by live superheated steam to pro-

gressively higher temperatures. The temperatures are adjusted to yield approximately the same volume of distillate from each still and to yield from the last still a residual pitch of m.p. at least 235° F. (146° C.) by the ball and ring test, which is suitable for use as fuel, either alone or mixed with powdered coal or the like.—L. A. C.

**Benzol mixtures; Preparation of — which remain liquid at low temperatures.** Tetralin G.m.b.H. G.P. 329,833, 29.3.16.

THE addition to benzol, with a low toluene content, of hydronaphthalenes consisting chiefly or wholly of "decahydronaphthalene," increases its energy without materially affecting the ignition point. The freezing point of a mixture of 90% of benzol and 10% of "decahydronaphthalene" is -3° C.; a 70:30% mixture freezes at -20° C.—W. J. W.

**Lubricants.** J. J. Hood, H. Spence, and P. Spence and Sons, Ltd. E.P. 153,922, 16.8.19.

A LUBRICANT consists of a suspension of aluminium powder, with or without the addition of other finely divided solids, such as mica or graphite, in a suitable carrier, such as oil, petroleum jelly, or solidified oil.—L. A. C.

**Mine waste [from collieries etc.]; Method of and apparatus for utilising — by means of the heat of dump-heaps.** W. Ostwald, Assr. to The Chemical Foundation, Inc. U.S.P. 1,369,350, 22.2.21. Appl., 21.7.16.

SEE E.P. 107,446 of 1916; J., 1917, 956.

**Peat; Method and means for removing water from —.** A. ten Bosch, Assr. to N. V. Nederlandsche Veenververkings Maatschappij. U.S.P. 1,369,811, 22.2.21. Appl., 3.10.18.

SEE E.P. 128,064 of 1918; J., 1919, 565 A.

**Coking oven.** O. Piette, Assr. to Soc. Anon. des Fours à Coke Semet-Solvay & Piette. U.S.P. 1,370,481, 1.3.21. Appl., 6.11.18.

SEE E.P. 127,165 of 1918; J., 1919, 493 A.

**Coke oven and like gases; Process for recovering ammonia from —.** Soc. Ind. de Prod. Chim. E.P. 127,549, 27.9.18. Conv., 27.5.18.

SEE U.S.P. 1,366,301 of 1921; J., 1921, 147 A. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 109,814 and 114,236; J., 1918, 545 A, 241 A.)

**Illuminating and other gases; Purification of —.** W. O. Felt. E.P. 159,905, 22.9.17.

SEE U.S.P. 1,237,767 of 1917; J., 1917, 1089.

**Oil-cracking stills; Process of condensing and treating distillates from —.** R. Fleming. E.P. 135,855, 26.11.19. Conv., 25.3.18.

SEE U.S.P. 1,325,868 of 1919; J., 1920, 149 A.

**Distillation of [mineral] oils or other fluids; Apparatus for —.** W. J. Mellersh-Jackson. From International Gasoline Process Corp. E.P. 159,774, 14.4.20.

SEE U.S.P. 1,340,532 of 1920; J., 1920, 510 A.

**Flow of gas in purifiers, condensers, and the like; Means for reversing the —.** Firth, Blakeley, Sons, and Co., Ltd., and W. Blakeley. E.P. 159,261, 15.11.19.

**Blast-furnace gas.** E.P. 148,802. See I.

**Gas-filter.** U.S.P. 1,368,540. See I.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

**Wood; Carbonisation of — by internal combustion engine exhaust gases.** L. Tissier. Chim. et Ind., 1921, 5, 136—142.

THE average loss of heat in the exhaust of internal combustion engines is 40%, the temperature of the gases being 400°—450° C. and their composition 78% N<sub>2</sub>, 14% CO<sub>2</sub>, and 8% O<sub>2</sub>. The author has designed a kiln in which such gases pass directly in contact with the charge of wood, heating it to 250° C. and effecting slow distillation. The presence of 7—8% oxygen in the gas is essential to satisfactory carbonisation. The kiln is an upright brick cylinder of 3—12 cub. m. internal capacity, provided with charging and discharging doors, a grate, and gas inlet and exit. One kiln is sufficient for engines up to 60—80 H.P. Condensers for the distillation products may be added. Carbonisation takes 12—24 hrs. In trials in Algeria with light wood cut 8—10 months a yield of 30—33% of charcoal was obtained and with drier material up to 40%. The charcoal is of good quality. The pyrolygneous acid contains 6.2% of acetic acid and 1.5% of methyl alcohol. Owing to its slow and perfectly regular action the process offers marked advantages over other methods of wood distillation in both yields and quality of product. The author advocates central plants for working up the crude pyrolygneous acid and the use of a part of the charcoal produced for feeding the gas producers generating gas for the engines. It is claimed that it will be more economical even for gas engines at a distance from a forest to transport wood, carbonise it as described, and use the charcoal than to carbonise by less efficient methods in the forest and to transport charcoal. It is calculated that the exhaust from the gas engines of over 25 H.P. running in France and Algeria alone would suffice to effect the world's total present distillation of wood.—C. I.

**Shale; Plant design for hot gas pyrolytic distillation of —.** L. Simpson. Chem. and Met. Eng., 1921, 24, 341—345.

IN a plant designed for the economical distillation of 2000 tons of shale per 24 hrs., the shale is crushed, screened, and passed over a magnetic roller. Uniform physical state of the shale before entering the retorts is secured by conveying the screened material through dryers heated by the products of combustion of the steam boilers after those products have been partially cooled in a gas pre-heater. The retorts are of vertical pattern, the upper portion diverging into two separate chambers into which shale is fed by a constant slow-feeding device. Four "double" retorts walled round with double steel plates, packed between with insulating material, form a "bench." Two discharging rollers at the lower end of a retort chamber cause a constant downward movement of shale, which is heated by the admission of pre-heated gases, the product of a previous operation. These gases are admitted into the chamber at several points and the supply can be controlled. It is claimed that the gas current increases the distillation effect by acting as a carrier for the oil vapours, and also as a diluent, lowering the vapour pressures of the volatile products. Light and heavier oils are condensed separately, and the cooling water from the heavy oil vapour condenser is fed directly to the steam boilers. Spent shale from the retorts is treated subsequently to recover the maximum yield of ammonia at the lowest cost.—C. A. K.

## PATENTS.

*Tar; Process for removing — from the products of the distillation of wood, coal, and the like.* E. Barbet et Fils et Cie. E.P. 158,915, 4.11.18. Conv., 3.11.17. Addn. to 120,558 (J., 1920, 57A).

THE gases issuing from the retorts are bubbled through tar to remove a portion of the tar from the gases, and then through cresol to extract the remainder of the tar. The apparatus is so constructed that loss of heat is reduced to a minimum, and the gases are at a temperature of 115°–130° C. as they pass through the cresol, whereby only tar is retained, and water and acetic acid leave the apparatus as vapour.—L. A. C.

*Distilling bituminous coal, brown coal, and shale by superheated steam; Process and apparatus for —.* M. Gercke. E.P. 159,246, 20.10.19.

THE coal or shale is subjected to the direct action of superheated steam whilst being continuously conveyed into and through a distilling chamber. The fuel is first preheated by conveying it through a steam-jacketed section or/and through an oil-bath heated to any desired temperature. The coal is conveyed by endless chains comprising articulated members or links formed with pistons which are maintained fluid-tight during their passage through the distilling chamber by labyrinth packings. The steam is conveyed in an unbroken circuit through the distilling chamber, a gas liquor collector, and the steam superheater. The oil bath for preheating the coal is situated in the lower part of the distilling chamber, the oil being circulated by means of a pump, through a heater heated by the waste gases from the steam boiler and also by the steam superheater.—A. G.

*Charcoal; Process for producing dense —.* L. F. Hawley, Assr. to The United States of America. U.S.P. 1,369,428, 22.2.21. Appl., 3.4.19.

CHARCOAL having a density of at least 0.95 is made by forming blocks of comminuted wood, subjecting them to a pressure of at least 30,000 lb. per sq. in., and then distilling them under a direct mechanical pressure, which varies periodically between 50 and 120 lb. per sq. in.—W. F. F.

*Illuminant [; Hydrogenated naphthalene as —].* K. Wimmer. G.P. 302,488, 23.7.15.

HYDROGENATED naphthalene, either alone or mixed with other illuminants, burns, e.g., in a paraffin lamp, with a flame of high luminosity. A suitable catalyst for the hydrogenation of naphthalene is prepared by reducing dry nickel formate or the like made to a paste with nr dissolved in oil. For the production of "decahydronaphthalene" the hydrogenation is effected at 180°–200° C. and 30 atm.—L. A. C.

*Ductile tungsten.* E.P. 155,851. See X.

## III.—TAR AND TAR PRODUCTS.

*Anthracene; Formation of — from benzene and ethylene.* J. E. Zanetti and M. Kandell. J. Ind. Eng. Chem., 1921, 13, 208–211.

ANTHRACENE is formed when benzene vapour and ethylene are passed through a heated quartz tube. The optimum temperature is 925° C., the yield being 0.675% of the benzene; above this temperature the yield decreases, and none is formed at 1000° C., whilst the amount of carbon produced increases.—W. P. S.

*Electrolytic reactions of naphthalene and its derivatives. 1. Electrolytic oxidation of naphthalene.* K. Ono. Nippon Kwagaku Kwai Shi (J. Chem. Soc. Japan), 1921, 42, 38–66.

NAPHTHALENE is oxidised electrolytically to  $\alpha$ -naphthoquinone, together with phthalic acid and a dark-brown resinous substance, in acid solution (mixtures of sulphuric acid and acetone or acetic acid), using a lead peroxide or platinum anode. A mixture of 100 c.c. of water and 20–30 c.c. of sulphuric acid (sp. gr. 1.84) is a suitable electrolyte. With increase of current density the amount of naphthalene oxidised increases, but the yield of  $\alpha$ -naphthoquinone does not increase proportionately; a suitable current density is 2 amp. per 100 sq. cm. for a lead peroxide anode and 3 amp. per 100 sq. cm. for a platinum anode. The amount of oxidation increases with rise of temperature, and the yield of naphthoquinone increases proportionally with a platinum anode, whereas with a lead peroxide anode the best results are obtained at room temperature. With increase in the quantity of electricity the yield of naphthoquinone diminishes, and secondary reactions take place with formation of a dark-brown substance. As oxygen-carriers, cerium nitrate, vanadic acid, potassium chlorate and chromate, manganous sulphate, potassium ferrocyanide, and chrome alum are effective in the above order.  $\alpha$ -Naphthol and a compound of  $\alpha$ -naphthol and naphthoquinone were detected in the reaction products. (Cf. J.C.S., Apr.)—K. K.

*Acetylene; Condensation of — with benzene and its derivatives in the presence of aluminium chloride.* O. W. Cook and V. J. Chambers. J. Amer. Chem. Soc., 1921, 43, 334–340.

BENZENE and its derivatives condense with acetylene in the presence of aluminium chloride to give  $\alpha\alpha$ -diarylethanes and derivatives of anthracene. Only a trace of styrene was found when benzene was condensed with acetylene. Certain derivatives of benzene, owing to preliminary reaction or condensation with aluminium chloride, do not act further with acetylene. (Cf. J.C.S., May.)—W. G.

*Phenol; Interaction of tin and —.* H. F. Zoller. J. Amer. Chem. Soc., 1921, 43, 211–212.

WHEN phenol is slowly distilled in the presence of tin it is partly decomposed, benzene and stannic oxide being formed. In one experiment about 30 c.c. of benzene was obtained from 200 g. of phenol. The reaction is practically negligible at the ordinary temperature, but is of importance in the manufacture of phenol and its use in serum laboratories.—W. G.

## PATENTS.

*Hydronaphthalenes; Manufacture of —. Purification of technical naphthalene for the manufacture of hydronaphthalenes.* Tetralin G.m.b.H. G.P. (A) 324,861, 25.2.15, (B) 324,862, 1.8.15, and (C) 324,863, 14.5.16.

(A) TECHNICAL naphthalene, after purification as described in (B) or (C), is heated to 150°–200° C. under a pressure of 10 atm. with sufficient hydrogen to produce the desired hydrogenated product. "Decahydronaphthalene" is not formed until the whole of the naphthalene has been reduced to tetrahydronaphthalene. Technical naphthalene is purified by agitation above 100° C. with a finely powdered or porous material, such as fuller's earth, kieselguhr, or animal charcoal, and (B) a finely divided or low-melting metal, such as nickel or sodium, or (C) an unstable compound of a metal with a non-acid residue, e.g., a metallic amide or carbide, such as sodamide or calcium carbide, or

with the metal or metallic compound alone, with subsequent distillation under reduced pressure.

—L. A. C.

*Methyleneanthraquinone and its substitution products; Preparation of* — K. H. Meyer. G.P. 330,550, 16.9.19.

THE compounds claimed, which serve as intermediates in the manufacture of vat dyes, are prepared by condensing anthranol or its substitution products with formaldehyde in alkaline or acid solution, with the exception of concentrated sulphuric acid. A solution of  $\alpha$ -hydroxyanthranol in hot 5% sodium hydroxide solution yields with formaldehyde a light-brown precipitate. A mixture of glacial acetic acid and concentrated hydrochloric acid may also be used as the condensing agent.—L. A. C.

*Benzol mixtures.* G.P. 329,833. See IIa.

*Illuminant.* G.P. 302,488. See IIa.

#### IV.—COLOURING MATTERS AND DYES.

*Indigotin; Constitution of* — R. Robinson. J. Soc. Dyers and Col., 1921, 37, 77—81.

THE case of indigotin is taken as an example of the manner in which partial valency formulæ may be employed to symbolise more accurately than is possible with expressions involving normal valencies only, the properties and relations of chemical individuals. Colour intensification by auxochromes is attributed to the making and breaking of partial valencies. Attention is drawn to the analogy between indigotin and indanthrene, and the probability that the cause of the colour resides in the interaction of the nitrogen atoms and the oxygen of the quinone groups is demonstrated by a consideration of the variations caused in each series by exchanging S and O for one or both of the NH groups. In both indigotin and indanthrene there is a double possibility of partial valency union between quinonoid oxygen and the auxochrome nitrogen, and the author's formulæ express this view of the origin of colour, and also fully symbolise the chemical properties of these compounds.

—F. M. R.

#### PATENT.

*Loke.* U.S.P. 1,369,252. See XIII.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Fabrics; Tensile, bursting, and ripping strain of* — J. Huebner. J. Soc. Dyers and Col., 1921, 37, 71—75.

THE resistance of a fabric to bursting or to ripping often affords valuable information, and in many cases these tests can replace the more tedious tensile test. Determinations of the tensile strain, carried out on hundreds of pieces of four-scoured plain cotton fabrics of different weights, indicate that when ascertaining the effect of chemical agents or processes upon the strength of a fabric, too much value should not be attached to average figures which are obtained from a small number of tests. The bursting strain of the fabric was determined by means of an apparatus of the Mullen type, a circular hole,  $\frac{1}{4}$  in. in diam. being punched in the middle of the test piece, as that permits the fabric to give way more readily along the line of least resistance than when a slit is used. Bursting tests with and without the central hole showed a constant ratio, and the former method was adopted owing to the increased life of the rubber diaphragm of the apparatus. The figures obtained show that

the bursting strain is comparable with the tensile strain, and that it affords a reliable indication of the strength of the fabric. A ripping test reveals any tendering or change more readily than does a tensile or bursting test. The warp or the weft threads are cut to a depth of 2 ins., and one end of the cut piece is fixed in the upper and the other end in the lower jaw of a Schopper paper-testing apparatus. The jaws are moved apart at a speed of 3 ins. per minute for a distance approximating to 2 ins., and the strain is applied at right angles to the threads to be broken. The figures show that the ripping strains of the lighter fabrics, when compared with those of the heavier fabrics, are proportionally much higher. Tests with plain fabrics, various matt weaves, and sateens, woven from the same quality and quantity of yarn, show that with the exception of the sateens the increased ripping and bursting strains as compared with those of the plain fabric are accompanied by decreased tensile strength. Bleaching increases the tensile strength slightly, but reduces the ripping strain, particularly in the warp way. Tannic acid increases the tensile strength considerably, but the ripping strain remains unchanged, except for a slight reduction in the warp way. The ripping strain is reduced by aluminium and iron mordants, but dyeing with direct and sulphide colours is without effect. The tensile strain is increased by a chromium mordant, but the bursting and ripping strains are reduced. On the whole the strength of linen fabrics is less regular than that of cotton fabrics of equal weights, and whilst the resistance to tensile and bursting strains is similar in each case, the ripping strain of a linen fabric is very much higher than that of a cotton fabric.

—F. M. R.

*Cellulose; Solubility of* — in the salts of the alkali and alkaline-earth metals. R. O. Herzog and F. Beck. Z. physiol. Chem., 1920, 111, 287—292.

THE solubility of cellulose in concentrated solutions of alkali and alkaline-earth salts (*cf.* Von Weimarn, J., 1912, 768) is a function of the hydration of the ions of the respective salt. The ions may be arranged in the following order:  $\text{NH}_4^+ < \text{K}^+ < \text{Na}^+ < \text{Li}^+ < \text{Ba}^{2+} < \text{Sr}^{2+} < \text{Ca}^{2+} < \frac{1}{2}\text{SO}_4^{2-} < \text{Cl}^- < \text{Br}^- < \text{I}^- < \text{CNS}^-$ . In experiments with raw and purified cotton, cotton wool, wood pulp, and hydrocellulose, no swelling or solution occurred in solutions of chlorides, bromides, nitrates, sulphates, thiocyanates, acetates, and lactates of sodium, potassium, ammonium, and magnesium, barium and strontium bromides and barium thiocyanate. Calcium nitrate attacked the cellulose. Swelling without disappearance of structure occurred in solutions of potassium iodide at 120°—140° C., and of alkaline-earth iodides at 120°—150° C. Solution of the cellulose took place in solutions of sodium iodide, lithium chloride, bromide, iodide, and nitrate, calcium iodide, bromide, and thiocyanate, strontium thiocyanate, and potassium mercuric iodide. "Commercial pure" calcium chloride will dissolve cellulose if the dilute solution be first neutralised with N/100 hydrochloric acid and then concentrated.—S. S. Z.

*Oralic acid from lignin etc.* Heuser and others. See XX.

*Methyl alcohol in sulphite spirit.* Sieber. See XVIII.

#### PATENTS.

*Spun material resembling wool or cotton; Producing a — from viscose solutions.* Glanzfäden A.-G. E.P. (A) 135,205, (B) 152,349, and (C) 152,350, 13.11.19. Conv., (A) 22.2.18, (B) 27.3.18, and (C) 6.4.18.

(A) A MATERIAL resembling wool or cotton, and not

possessing the high gloss of artificial silk, is prepared by saturating cellulose with an 18% solution of sodium hydroxide, and pressing out excess liquor until the mixture contains about 2 pts. of solution to 1 pt. of cellulose. After treatment with the alkali for not more than 12 hrs., the product is treated with carbon bisulphide, dissolved in weak sodium hydroxide solution, and "spun" in a mineral acid bath before any ripening can set in. (b) If the cellulose has been well opened up by thorough boiling during its preparation, the time of treatment with alkali can be reduced to 10 mins. for a flocky, or 2 hrs. for a matted material. (c) The mixture of cellulose and sodium hydroxide solution is pressed out until it contains not more than 2 pts. of solution to 1 pt. of cellulose, and the temperature of the mixture is not allowed to rise above 20° C. before treatment with carbon bisulphide.—L. A. C.

*Vulcanised fibre: Softening hard* —. C. Hasenbring. G.P. 329,891, 21.12.17.

THE material is soaked in an emulsion of equal parts of magnesium or calcium chloride solution and wood tar.—L. A. C.

*Woollen fabrics; Method of protecting* — from moths. E. Naefe. E.P. 160,039, 15.1.20.

SEE G.P. 304,506 of 1917; J., 1918, 409 A.

*Vegetable fibres etc; Process for scouring* —. E. T. J. Watremez. U.S.P. 1,370,076, 1.3.21. Appl., 29.10.19.

SEE E.P. 139,457 of 1919; J., 1920, 686 A.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Mercerisation; Use of caustic potash in* —. E. Ristenpart. Textilber., 1921, 2, 130—131.

COTTON was immersed without tension for 5 mins. in a caustic alkali solution, mangled, washed three times in water, immersed in water over-night, soured with acetic acid, and dried at the ordinary temperature. Comparative tests with solutions of sodium and potassium hydroxides showed that the sodium and potassium alkali-cellulose compounds have similar properties. In very concentrated solutions, caustic soda (owing to its greater viscosity) has less mercerising effect than caustic potash, but under the usual conditions 1 g.-mol. of caustic soda is equal to 1½ g.-mols. of caustic potash.—A. J. H.

*Crêpe-effects; Production of* — on cotton by mercerisation. A. Liebmman. Sealed Note, No. 1478, deposited 24.5.04. Bull. Soc. Ind. Mulhouse, 1920, 86, 484. Report by T. Baumann, *ibid.*, 485.

FABRIC which has previously been boiled-out, bleached or mercerised under tension, and dried, is "wetted-out," calendered to remove excess of moisture, dried unevenly according to a desired design by means of hot rollers or plates, mercerised with caustic soda, rapidly rinsed, soured, and washed. The caustic soda affects only the wet parts of the fabric, so that brilliant crêpe-effects are obtained. Baumann reports that satisfactory results are obtained with a fabric previously boiled in a weak solution of sodium carbonate and well bleached. Fabrics boiled with caustic soda give unsatisfactory results.—A. J. H.

*Silk; Method for weighting and dyeing* —. A. Tiska. Textilber., 1921, 2, 128—129.

SILK is prepared by steeping it in water at 40° C. for 1 hr., boiling in a 30—35% soap solution, wash-

ing, and souring with hydrochloric acid. The silk is weighted 80—100% by immersing it four times alternately in stannic chloride solution of 80° B. (sp. gr. 2.43) and disodium phosphate solution of 7° B. (sp. gr. 1.052) and washing after each immersion. The weighted silk is treated with a warm soap solution for 1 hr., and without washing is dyed black in a bath containing 100% (on weight of silk) of soap and 100% of "logwood crystals." The temperature of the bath is raised from 62° to 75° C. during ½ hr., 5% of aniline is added, the temperature is increased to 92° C. during ½ hr., and so maintained for 1 hr. The dyed silk is well washed, treated for ½ hr. in a warm boiled-off soap bath to which acetic acid and 0.3% of Water Blue have been added, and finally brightened with oil and citric acid.—A. J. H.

*Cellulose; Behaviour of highly-oxidised* — towards direct cotton colours. E. Knecht. J. Soc. Dyers and Col., 1921, 37, 76—77.

THE behaviour of the oxycellulose previously described (J., 1920, 718 A) towards a number of direct cotton colours has been examined quantitatively. A known weight of the oxycellulose powder was heated for 1 hr., under a reflux condenser, with an excess of the dye solution in presence of sodium hydroxide, the solution then filtered, and the dye remaining in the filtrate titrated in the usual manner. The dye was destroyed by the oxycellulose in an amount which, in some cases, largely exceeded the maximum quantity employed for a full shade in dyeing, but the oxycellulose remained undyed, although its copper number was reduced from 11 to 0.9. Prolonged boiling with the azo dye removed the oxycellulose completely and the remaining cellulose was dyed. The dyeing properties of the oxycellulose can be restored by treatment with sodium hydroxide alone, but the concentration necessary is greater than that used in the dyeing experiments. Direct cotton colours of the Primuline type, which are not affected by reducing agents, dye the highly oxidised cellulose immediately. Such a highly oxidised cellulose is never met with in practice, but the results obtained indicate that the presence of even small amounts of oxycellulose in cotton goods may result in an appreciable loss of dyestuff during dyeing.

—F. M. R.

*Ice colours; Developers for* —. O. and E. Schlein. Sealed Notes Nos. 1681 and 1743, deposited 5.12.06 and 21.5.07. Bull. Soc. Ind. Mulhouse, 1920, 86, 486—489. Report by P. Binder, *ibid.*, 489—493.

IN the formation of Para Red, solutions of diazotised *p*-nitraniline containing an excess of mineral acid give pale yellowish shades. To obtain rich and bluer shades the free acid must be neutralised with an excess of sodium acetate. A satisfactory and cheaper process consists of replacing part of the sodium acetate by its equivalent of sodium carbonate, in quantity just insufficient completely to neutralise the mineral acid. For the preparation of printing pastes containing diazotised *p*-nitraniline it is preferable to replace all the sodium acetate by its equivalent of caustic soda. Binder reports favourably on the process, although slightly inferior shades are obtained than when sodium acetate is used alone. When disodium phosphate, calcium carbonate, or magnesium carbonate is used in place of sodium carbonate, still less satisfactory results are obtained. In a process communicated by C. Sunder, the diazo solution contains:—2760 g. of *p*-nitraniline, 1500 g. of sodium nitrite, 10,600 g. of hydrochloric acid of 18° B. (sp. gr. 1.143) and 36 l. of calcium acetate solution of 15° B. (sp. gr. 1.116) per 120 l.—A. J. H.



*Paramine Brown; Production of discharges on —.* H. Schmid. Sealed Notes Nos. 1504 and 1539, deposited 9.11.04 and 27.12.05. Bull. Soc. Ind. Mulhouse, 1920, 86, 477–479. Report by C. Sunder, *ibid.*, 481–482.

Good white discharges on a Paramine Brown ground are obtained if the fabric is padded with a solution containing per l., 30–40 g. of *p*-phenylenediamine dihydrochloride, sodium acetate (sufficient to neutralise 50–75% of the hydrochloric acid present), 25–30 g. of sodium chlorate, and 1–10 mg. of vanadium chloride (added just before use), dried, printed with a discharge paste containing hydrosulphite NF, steamed in the Mather-Platt, and washed. For coloured discharges basic dyes are used in the discharge paste and are subsequently fixed with tannic acid. Pure white discharges are also obtained by means of a colourless solution containing per l., 16 g. of *p*-phenylenediamine, 20 g. of ammonium chloride, 20 g. of sodium chlorate, 20 g. of potassium ferrocyanide, and 1.5 g. of Rongalite C, which has been filtered after standing for several hours. The fabric is padded in the solution, dried, printed with a discharge paste containing sulphites or hydrosulphite-formaldehyde and steamed in the Mather-Platt. Coloured discharges may be obtained by the usual methods employed with "prussiate" Aniline Black. Sunder reports that discharges obtained by the first process are not satisfactory, but that this may be due to the impurities present in the *p*-phenylenediamine. Light shades produced by the "prussiate" process appear bronzy and require correction by means of Xanthochromine, Naphthene S, or Fuscamine. No precipitation takes place in the padding mixture when free *p*-phenylenediamine or its acetate is used.—A. J. H.

*Discharges on fabrics having a ground formed by the oxidation of an aminophenol; White and coloured —.* H. Schmid. Sealed Note No. 1517, deposited 30.12.04. Bull. Soc. Ind. Mulhouse, 1920, 86, 479–480. Report by C. Sunder, *ibid.*, 483.

MONO-AMINOPHENOLS, diaminophenol (amidol), and *o*-anisidine may be oxidised on textile fibres to give brown to dark brown shades, capable of being discharged. The fabric is padded with a solution containing per l., either 40 g. of *p*-aminophenol hydrochloride, 5 g. of acetic acid (50%), 30 g. of potassium ferrocyanide, 25 g. of sodium chlorate, and 4–5 g. of crystalline sodium acetate; or 40 g. of *o*-aminophenol, 40 c.c. of hydrochloric acid of 20° B. (sp. gr. 1.162), 30 g. of potassium ferrocyanide, and 25 g. of sodium chlorate, then dried, printed with a discharge paste containing a mixture of sodium sulphite and sodium acetate, steamed in the Mather-Platt, washed, and soaped. Coloured discharges may be obtained in the usual way with basic and pigment colours. Sunder reports that shades produced from *o*-aminophenol lack depth. The brown shade produced from *p*-aminophenol is somewhat green, and is therefore suitable for correcting the red tone of Paramine Brown. *m*-Aminophenol yields a shade without value.

—A. J. H.

*Diastase preparations; New — and their importance in the textile industry.* G. Tagliani. Z. angew. Chem., 1921, 34, 69–73.

Two preparations, "Fermasol DS" and "Fermasol DB" have been placed on the market recently by the Swiss Ferment Co.; they are rich in animal amylase, liquefy starch rapidly, do not attack or weaken cotton fibres, and possess other advantages over the usual diastatic preparations used in the textile industry. Comparative experiments with these and other diastatic preparations are described.

—W. P. S.

#### PATENT.

*Albumins [protalbinic and lysalbinic acids]; Use of decomposition products of — [in dyeing].* K. Bennert. G.P. 330,133, 25.12.15.

THE addition of these acids or their salts to dye-baths, *e.g.*, of vat dyes, produces more level and deeper and brighter shades.—L. A. C.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Sulphuric acid; Supply of nitrogen oxides in the manufacture of — by the chamber process.* S. Pagliani. Giorn. Chim. Ind., 1919, 1, 57–61.

THE author discusses this question, mainly in connexion with the work of Fairlie (J., 1917, 196; 1918, 632 A), Goldmann (J., 1919, 251 A), and Chrisp (Chem. Trade J., 1919, 64, 91).—T. H. P.

*Sulphur dioxide and sulphur trioxide; Determination of — in burner gases and oleum.* A. Sander. Chem.-Zeit., 1921, 45, 261–263.

A METHOD of estimating sulphur dioxide is based on its conversion by means of sodium hydroxide into bisulphite, and the reaction of the latter with mercuric chloride to form a double salt, with liberation of hydrogen chloride; neutralisation then demands an amount of sodium hydroxide equivalent to that used for the conversion of the sulphur dioxide. 10 c.c. of *N*/10 sodium hydroxide is introduced, together with some methyl orange as indicator, into an absorption flask, provided with a bottom run-off tap. By means of an aspirator, the burner gases are slowly drawn in until neutralisation takes place, and from the known volume of the gases the total sulphur dioxide and trioxide is calculated. The solution is then drawn off, treated with a saturated solution of mercuric chloride, and titrated with *N*/10 sodium hydroxide, the result enabling the amount of sulphur dioxide to be determined; sulphur trioxide is calculated by difference. Estimation of sulphur dioxide and trioxide in oleum is carried out in a similar manner.—W. J. W.

*[Sulphuric acid;] Gaillard towers [for concentrating —] and the electrical condensation of acid fumes.* M. Kaltenbach. Chim. et Ind., 1921, 5, 143–149.

THE ordinary Gaillard tower system consists of the tower proper, which the hot gases enter at 850° C. and leave at 200°–220° C.; a small tower fed with weak acid from the scrubbers and acting as a recuperator, and the coke scrubbers. For satisfactory work the gas should leave the small tower at a temperature just above its saturation point, say 120°–130° C. Assuming that a true static equilibrium exists throughout the apparatus, the concentration of liquid acid in equilibrium with the vapour varies directly with the temperature. Though such a perfect equilibrium is not obtained, experiments are described to show that it is approached. No process can therefore at once give strong condensed acid and a complete condensation, and the function of the Cottrell process, which cannot affect the equilibrium between the liquid and gaseous phases, is limited to the aggregation of the vesicles of liquid formed at the moment of condensation. The author considers that rationally designed coke scrubbers can perform this more cheaply than electrostatic precipitators. A slightly lower acid content in the exit gases for the same temperature with the use of the Cottrell process is attributed to the oxidation of sulphur dioxide in the latter case by nitrous gases derived from incompletely denitrated acid. The author suggests the use of two small coke scrubbers each preceded by an efficient cooling coil and having a brick grid beneath which the gas enters, the coke increasing in fineness from bottom



to top, and with no heavy timbers to obstruct cooling. The first scrubber is to condense acid down to the lowest strength at which it can be economically concentrated, the product of the second being run to waste.—C. I.

**Reaction  $3\text{HNO}_3 \rightarrow 2\text{NO} + \text{HNO}_2 + \text{H}_2\text{O}$ ; Condition of an unattackable electrode in the —.** A. Klemenc. Z. Elektrochem., 1921, 27, 110—112.

By comparing the observed with the thermodynamically calculated potentials, when a platinum electrode is in contact with the system nitric acid—nitrous acid—nitric oxide at equilibrium, it is shown that the reaction indicated is  $\text{NO} + 2\text{H}_2\text{O} \rightarrow 4\text{H}^+ + \text{NO}_3^-$  in solutions about 3N, but in dilute solutions the reaction  $\text{NO} + \text{H}_2\text{O} + 2\text{H}^+ + \text{NO}_2^-$  also comes into play (cf. Pick, J., 1920, 446 A). (Cf. J.C.S., Apr.)—E. H. R.

**Phosphoric acid and soluble phosphates; Investigation of the U.S.P. assay for —.** A. E. Stearn, H. V. Farr, and N. P. Knowlton. J. Ind. Eng. Chem., 1921, 13, 220—225.

THE U.S.P. method of determining phosphoric acid (neutralisation with sodium hydroxide, using phenolphthalein as indicator, precipitation with an excess of silver nitrate, neutralising the mixture with zinc oxide, and titrating the excess of silver) is untrustworthy except at one definite concentration, namely 6.2 mg. per c.c. The error is probably due to the formation of slightly soluble acid phosphates of silver, and the authors therefore recommend the following process, in which the phosphoric acid is first converted into the tri-sodium salt:—10 c.c. of a 1% solution of the sample is treated with 5 g. of sodium nitrate, cooled in an ice bath, and titrated with N/1 sodium hydroxide solution, using phenolphthalein as indicator. The number of c.c. of alkali required is divided by two, and this quantity is also added to the mixture. The latter is then treated with 50 c.c. of N/10 silver nitrate solution, diluted to 100 c.c., filtered, and the excess of silver titrated in an aliquot portion of the filtrate.—W. P. S.

**Caustic soda; Preparation of — from sodium sulphate.** B. Neumann and E. Karwat. Z. Elektrochem., 1921, 27, 114—124.

To determine the course of the reaction between sodium sulphate and lime and the maximum yield of caustic soda to be expected, experiments were made at 15°, 40°, 70°, and 100° C. at different dilutions starting with sodium sulphate and lime on the one hand and calcium sulphate and sodium hydroxide on the other. To obtain a condition of equilibrium, continuous shaking for several days was necessary. The highest yield, 60%, was obtained at the lowest temperature, 15° C., and the greatest dilution, N/10, the corresponding yield at 100° C. being only 27%. The yield falls rapidly as the temperature or concentration is increased. On account of the high dilution required, the process is of no technical interest. A further drawback is that, during concentration, reaction takes place between the caustic soda and calcium sulphate, lime being precipitated with a corresponding loss of caustic soda. The yield is not increased by working under pressure.—E. H. R.

**Potassium in silicates; Method for the determination of —.** J. J. Morgan. J. Ind. Eng. Chem., 1921, 13, 225—227.

About 0.5 g. of the silicate is decomposed with hydrofluoric acid, the mixture evaporated to dryness, the residue dissolved in 25 c.c. of 3N hydrochloric acid, an excess of 10% perchloric acid added, and the mixture evaporated until fumes of perchloric acid are evolved; the residue is treated with

water and again evaporated with the addition of perchloric acid, and this is repeated until the residue dissolves completely in hot water. After a final evaporation, the residue is treated with 20 c.c. of 98% alcohol (containing 1 c.c. of 60% perchloric acid per 300 c.c.), stirred during 15 mins., the potassium perchlorate then collected on a filter, washed with the alcohol (using about 40 c.c.), dried at 130° C., and weighed. Calcium and sodium sulphates interfere, and if these salts are present, the sulphate must be determined in the weighed precipitate.—W. P. S.

**Adsorption of alkali chlorides by animal charcoal.** H. Hartleben. Biochem. Zeits., 1921, 115, 46—51.

ALL alkali chlorides are adsorbed by animal charcoal to the same extent within the limits of experimental error.—S. S. Z.

**Potassium perchlorate; Preparation of —.** E. Blau and R. Weingand. Z. Elektrochem., 1921, 27, 1—10.

POTASSIUM perchlorate is best prepared by heating potassium chlorate in a quartz flask at 430° C. without a catalyst for 8 hrs. In this way 69 g. of perchlorate can be obtained from 100 g. of chlorate. There is always about 3% of the chlorate undecomposed, but this is removed by a single crystallisation. Iron vessels are unsuited for this preparation. Potassium perchlorate can also be obtained by electrolysis of solutions of potassium chlorate between a nickel cathode and a platinum anode, using a current density of 0.15 amp. per sq. dm. The process suffers from the drawback that, even with a rapidly circulating electrolyte, the electrodes become incrustated with the sparingly soluble perchlorate. This difficulty is surmounted by electrolyzing sodium chlorate and converting the very soluble sodium perchlorate into the potassium salt by treating with potassium chlorate, thus regenerating sodium chlorate which is used in the next electrolysis. At 27° C. the current yield of the process increases with increasing cathode current density, but a change in the anode current density has no effect on the yield. The electrolysis of barium chloride solution at 60° C. under the same conditions gives a current yield of 50—60% of barium perchlorate. (Cf. J.C.S. May.)—J. F. S.

**Polysulphide-sulphur; Determination of —.** A. Wöber. Z. angew. Chem., 1921, 34, 73.

TEN c.c. of the diluted polysulphide solution is added to an excess of 10% sodium sulphite solution, the mixture heated at 50° C. for 15 mins., cooled, and the thiosulphate formed by the interaction of the polysulphide and the sulphite is determined as described by Bodnar (J., 1915, 1111). Allowance must be made for any thiosulphate present originally in the polysulphide solution, and if the latter contains alkali carbonate, the thiosulphate determinations must be made after the solution has been treated with cadmium acetate or zinc sulphate, and filtered.—W. P. S.

**Chromic oxide; Carrying down of lime and magnesia by precipitates of —.** E. Toporescu. Comptes rend., 1921, 172, 600—602.

WHEN chromium is precipitated by ammonia from solutions of its salts containing calcium or magnesium salts, the amounts of calcium or magnesium carried down increase with the concentration of their salts, and tend towards limits corresponding to the chromites,  $\text{Cr}_2\text{O}_3 \cdot 3\text{CaO}$  and  $\text{Cr}_2\text{O}_3 \cdot 3\text{MgO}$ , respectively. The calcium or magnesium may be removed from such precipitates by washing them on the filter or by decantation with a boiling 5% solution of ammonium nitrate.—W. G.

*Nitrogen; Cathodic reduction of dissolved elementary* — E. Tiede and A. Schleede. Z. Elektrochem., 1921, 27, 112–114.

IN 1807 Davy claimed to have obtained nitric acid and ammonia by the electrolysis of distilled water containing dissolved air, but Rayleigh failed to confirm this result. The authors have repeated the experiment under widely varying conditions, at pressures from atmospheric up to 150 atm., and have failed to obtain a trace of ammonia.—E. H. R.

*Sulphur; Elastic* — T. Iredale. Kolloid-Zeits., 1921, 28, 126–127.

ELASTIC sulphur is prepared by treating powdered sodium thiosulphate with one-half its weight of concentrated nitric acid, and after the reaction is complete, adding water and washing the product. A yellow elastic transparent mass is obtained which after distension to four times its length will regain its original form. Cooling to 0° C. makes it opaque and brittle, but heating in water to 100° C. reproduces the elastic characteristics. After 24 hrs. it passes completely into the crystalline form.

—J. F. S.

*Heat of dissociation of iron pyrites.* Kamura. See X.

*Mixed and waste acids.* Toussaint. See XXII.

*Arsenic and phosphoric acids.* Desbourdeaux. See XXIII.

#### PATENTS.

*Oxides of nitrogen; Production of — and catalyst therefor.* General Chemical Co., Assees. of W. W. Scott. E.P. 136,158, 2.12.19. Conv., 7.9.18.

THE catalyst consists of a mixture of cobaltic and aluminium oxides in the proportion of 100 pts. Co to 2.5–30.0 pts. Al. It is prepared by evaporating a solution of the nitrates and igniting the residue. The material is broken up, and fragments passing through an 8-mesh sieve but retained on a 20-mesh sieve are used. With an air-ammonia mixture of 8.3% NH<sub>3</sub> by vol., a conversion of 84.5% is obtained, or, if the catalyst is prepared at a temperature not exceeding 650° C., as high as 94%. The catalyst has a longer life and is less sensitive to dust than any other metallic oxide catalyst. It may also be used on a carrier, such as pumice.

—C. I.

*Alkali pentaborates; Preparation of — direct from boron ores.* A. A. Kelly and B. D. Jones. E.P. 153,992, 15.11.19.

BORON ores are mixed with sufficient alkali to ensure a proportion of 1 mol. of Na<sub>2</sub>O or K<sub>2</sub>O per 5 mols. of B<sub>2</sub>O<sub>3</sub>, and the mixture is calcined, and treated with carbon dioxide in presence of water, either under atmospheric or increased pressure. After concentration of the liquor, the alkali pentaborate is crystallised out.—W. J. W.

*Aluminium oxide; Process of producing — from chloride of aluminium.* S. E. Sicurin. E.P. 159,086, 19.1.20.

ALUMINOUS raw material is dissolved in hydrochloric acid, and the solution is saturated with hydrogen chloride, introduced at the top of the vessel. The acid mother liquor is then displaced from the precipitated crystals of aluminium chloride by forcing in a concentrated solution of aluminium chloride at the bottom of the vessel, after which this solution is separated from the crystals by centrifugal action, and the latter are calcined. The mother liquor may be used for solution of further raw material. To avoid accumulation of iron in the liquor, it is treated with hot

sulphuric acid (sp. gr. 1.71), the gaseous hydrogen chloride evolved being utilised in the initial process; the sulphuric acid solution is concentrated and cooled, and the precipitated iron sulphate separated.—W. J. W.

*Sulphurous acid; Process of and apparatus for the manufacture of liquid — from dilute sulphurous acid gas.* P. Pascal. E.P. 159,337, 2.12.19.

DILUTE gas from pyrites or blende burners is compressed, cooled, and washed in a tower with carbolic oil of sp. gr. 1.0 obtained in the distillation of coal tar between 175° C. and 225° C. This absorbs one-fourth of its weight of sulphur dioxide at atmospheric temperature and pressure. The oil passes on through a heat interchanger and is distilled under reduced pressure, a plate column removing any oil driven over with the gas; the latter is subsequently liquefied by compression.

—C. I.

*Chromous chloride; Process of making* — M. C. Taylor, Assr. to N. D. Baker. U.S.P. 1,369,204, 22.2.21. Appl., 15.12.19.

A solution of chromic chloride containing 1–2% of free hydrochloric acid is electrolysed in a cell having a diaphragm.—C. I.

*Pickling solutions; Method of and apparatus for reclaiming spent* — H. S. Marsh and R. S. Cochran. U.S.P. 1,369,451, 22.2.21. Appl., 9.1.20.

THE salts in the hot solution are precipitated by cooling, heat being exchanged with a counter current of the cooled solution.—C. I.

*Zinc chloride; Production of anhydrous* — P. Danckwardt, Assr. to The Danckwardt Process Co. U.S.P. 1,369,729, 22.2.21. Appl., 23.10.19.

A mixture of aluminium chloride, carbon, and zinc ore is gradually heated, the zinc chloride formed being volatilised and condensed.—C. I.

*Ammonium phosphate; Method of making* — H. C. Hetherington and J. M. Braham, Assrs. to United States of America. U.S.P. 1,369,763, 22.2.21. Appl., 10.6.20.

MONO-AMMONIUM phosphate is crystallised from slightly acid solution at a temperature above 105° C.—C. I.

*Ammonium salts; Preparation of* — Akt.-Ges. für Stickstoffdünger. G.P. (A) 299,141 and (B) 300,141, 5.7.16.

(A) CALCIUM cyanamide, which has previously been treated with carbon dioxide, is heated to temperatures above 100° C., under pressure, in presence of salts which decompose ammonium carbonate, such as those of the alkaline earths, and of water or steam. The process may be carried out in presence of catalysts, such as manganese dioxide and ferric hydroxide, which assist the formation of urea. (B) Dicyanodiamide, or a solution containing it obtained by treating crude calcium cyanamide with water, may be used as initial material.—W. J. W.

*Potassium salts; Apparatus for effecting continuous solution of crude* — G. Sauerbrey, Maschinenfabrik A.-G. G.P. 328,448, 6.2.20.

THE apparatus consists of two rotary drums, placed one behind the other, and each provided with a partition mounted on a support which passes through both drums and is suspended from external brackets. Each drum is divided by the partition into two compartments, in which the solvent and the solid material move respectively in the same and in opposite directions.—W. J. W.

**Alkali carbonates; Manufacture of** — from alkali sulphides and hydrosulphides. P. Beck. G.P. 329,832, 25.7.17.

ALKALI sulphides and hydrosulphides are converted into alkali carbonates by the action of magnesium bicarbonate, or of carbon dioxide in the presence of magnesium carbonate, oxide, or hydroxide, in aqueous solution.—L. A. C.

**Potassium ferrocyanide; Manufacture of** — [from calcium cyanamide]. Strontian- und Potasche-Fabr. Rosslau, Zweigniederlassung der Dessauer Zucker-Raffinerie G.m.b.H. G.P. 330,194, 15.2.20.

A MIXTURE of, e.g., 100 pts. of calcium cyanamide, 75 pts. of potassium carbonate, and 30 pts. of iron filings is heated to redness in the absence of air; the product is cooled, ground to a powder, and extracted with water.—L. A. C.

**Cooling and washing tower for gas from pyrites kilns.** O. Froberg. G.P. 330,650, 30.12.19.

THE tower is built of acid-resisting ferro-concrete and is provided with a firebrick roof carrying the system of spraying tubes, each of which is attached separately to the roof, and all of which are connected with a central supply tube. The interior of the tower is filled with small glazed earthenware tubes, and a protective acid-resisting or impermeable sheath may be arranged, if necessary, round the inner walls.—A. R. P.

**Alkali phosphate; Process for the preparation of** —. Chem. Fabr. Rhenania A.-G. G.P. 330,840, 4.2.17.

NATURALLY occurring phosphates, e.g., vivianite or triphosphate, are heated with solid alkali carbonate to 700°–800° C. Extraction of the sintered mass with water yields a solution of alkali phosphate.

—A. R. P.

**Lining for calcium cyanamide furnaces.** G. Polysius. G.P. 330,943, 6.9.19.

THE furnace is lined with quartzite, cement, or concrete, with or without an admixture of cement clinker. No reaction takes place between the charge and the lining, nor does the charge stick to it.—A. R. P.

**Arsenic and sulphur; Separate production of** — from sulphur ores containing arsenic. A. Wyporek, and Rheinisch-Nassauische Bergwerks- und Hütten-A.-G. G.P. 331,063, 7.8.19.

THE ore is heated rapidly to a high temperature, whereby the arsenic is volatilised with very little loss of sulphur, and the ore can then be roasted to produce gases from which sulphuric acid almost free from arsenic is obtained.—L. A. C.

**Zinc oxide; Manufacture of** —. New Jersey Zinc Co., Assees. of J. A. Singmaster, F. G. Breyer, and A. E. Hall. E.P. 137,513, 24.11.19. Conv., 8.1.19.

SEE U.S.P. 1,322,088 of 1919; J., 1920, 22 A.

**Oxides and other compounds of metals; Production of metal** —. New Jersey Zinc Co., Assees. of F. G. Breyer, A. E. Hall, and G. R. Waltz. E.P. 147,530, 24.11.19. Conv., 12.7.19.

SEE U.S.P. 1,322,089–90 of 1919; J., 1920, 22 A.

**Oxides and other compounds of metals; Production of metal** —. New Jersey Zinc Co., Assees. of J. A. Singmaster, F. G. Breyer, and E. H. Bunce. E.P. 149,927, 26.11.19. Conv., 22.7.19.

SEE U.S.P. 1,322,143 of 1919; J., 1920, 22 A.

**Separating aqueous vapours from liquids and preparation of dilute sulphuric acid.** E.P. 159,054. See I.

**Filling material for reaction chambers.** G.P. 330,226. See I.

**Tin-pot skimmings.** E.P. 158,926. See X.

**Metallic oxides and sulphur dioxide.** G.P. 331,177. See X.

## VIII.—GLASS; CERAMICS.

**Glass-house furnaces; Design and operation of** —. F. J. Denk. J. Amer. Ceram. Soc., 1921, 4, 224–236.

THE proper design of regenerator chambers is discussed and two inefficient designs are criticised. The gases being heated should move upwards, and those being cooled should pass downwards. The position of the ports should be selected carefully and losses by heat radiation minimised as much as possible. The reversing valve should be efficient and operated regularly, and the furnace should be fitted with thermo-couples and draught recorders. Analyses of the waste gases should be made frequently, and the operation of the furnace controlled accordingly.—H. S. H.

**Glasses; Simple expansibility test for determining the welding properties of** —. D. E. Sharp. J. Amer. Ceram. Soc., 1921, 4, 219–223.

THE ends of two glass rods are heated and welded together by forceps and a thread pulled out with the two glasses forming opposite sides. If the glasses have different coefficients of expansion the strip bends into a curve, the glass of higher coefficient of expansion being on the inside of the curve. The coefficient of expansion can be determined, if standard glasses of known coefficient are available, by finding the glass with which the specimen will give a thread without curvature on cooling.—H. S. H.

**Kaolinite; Study of the heating and cooling curves of Japanese** —. S. Satoh. J. Amer. Ceram. Soc., 1921, 4, 182–194.

HEATING and cooling curves up to 1400° C. were taken for Japanese kaolinite by means of a differential couple using quartz sand as standard. A heat absorption due to evaporation of moisture occurred up to 100° C., and was followed by a weak heat evolution from 100° C. to 300° C., possibly due to the oxidation of foreign materials. Heat was absorbed from 450° C. to 650° C. owing to dehydration, and a further endothermic change took place from 650° C. to 750° C., and was ascribed to the dissociation of kaolinite into free alumina and free silica. An exothermic reaction near 950° C. was considered to result from the polymerisation of alumina, while a heat evolution between 1200° and 1300° C. was ascribed to the formation of sillimanite from the dissociated free alumina and silica. The loss in weight on ignition occurred chiefly between 400° and 650° C., and the rate of increase per degree reached a maximum about 460° C. Changes in the microstructure of the kaolinite occurred at 600°, 900°–1000°, 1250°–1300°, and at 1400° C., when sillimanite began to develop. Heating and cooling curves for alumina prepared by calcining the nitrate, hydroxide, and sulphate gave exothermic reactions ascribed to the polymerisation of alumina at 800°–900° C. and at 1100°–1200° C., instead of at 950° and 1250° C., as in the case of kaolinite. In the discussion it was pointed out that some of the author's conclusions are erroneous, owing to his failure to recognise the endothermic change in quartz at 575° C.

—H. S. H.

**Fire-brick made from ganister, flint clay, and plastic clay mixtures; Tests of** —, with special reference to spalling. R. M. Howe and M. Sheppard. J. Amer. Ceram. Soc., 1921, 4, 206–212.

HAND-MADE bricks were prepared from mixtures of

ganister, flint clay, and plastic clay so as to vary the silica content from 53 to 77% and the alumina content from 43 to 20%. The fusion points of the bricks decreased gradually as the silica content increased, and were usually about 2 cones lower than the fusion points of the corresponding pure alumina-silica mixtures. It was found that behaviour under load at high temperatures was not a function of chemical composition, but depended more on the temperature of burning. The resistance to spalling of each brick (as tested by alternate heating and dipping in cold water) decreased on burning at higher temperatures. The bricks with the highest silica content were most resistant to spalling at the lower burning temperatures, but this advantage was lost at 1400° C.—H. S. H.

*Electrical porcelain; Effect of the replacement of free silica by alumina and zirconia in —.* R. Twells, jun., and C. C. Lin. J. Amer. Ceram. Soc., 1921, 4, 195–205.

In order to test whether free silica is detrimental to the mechanical and dielectric strength of electrical porcelain, a series of sixteen batches was prepared in which various proportions of the free silica were replaced, weight for weight, by alumina, zirconia, or combinations of both, and bars and discs, after burning at cone 8½–9 or at cone 12, were tested for shrinkage, transverse strength, impact strength, heat resistance, porosity, and dye penetration. When zirconia replaces flint overburning of the porcelain occurs at a lower temperature, but the ability to withstand sudden temperature changes is greatly increased. The firing shrinkage is increased, the colour is poorer, while the transverse strength is highest with equal parts of zirconia and flint. The maturing temperature of the porcelain is greatly raised by replacing flint by calcined alumina, but the ability to withstand sudden temperature changes is greatly decreased. The firing shrinkage is also decreased, and a high transverse strength is developed when the body approaches vitrification. The presence of free silica increases the dielectric strength.—H. S. H.

#### PATENTS.

*Dryer and kiln; Superposed —.* S. Knipfer, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. 1,369,235, 22.2.21. Appl., 23.4.20.

A continuous dryer is provided above a continuous tunnel kiln, with its exit end adjacent the entrance end of the kiln. Cars carrying the ware move successively through the dryer and kiln.—H. S. H.

*Tunnel kiln and method of operating same.* P. d'H. Dressler, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. 1,369,330, 22.2.21. Appl., 23.2.20.

The atmosphere formed by a hot body of gas in a central portion of the kiln is maintained without substantial movement except for convection currents. Gas is blown into the end of the kiln along the top of the kiln chamber so as to create a downward flow of the kiln atmosphere between the end and centre of the kiln, with a return flow along the bottom of the kiln to the kiln end where the excess gas escapes. The maintenance of an unduly high top temperature in the end of the kiln by reason of the convection current circulation of the kiln atmosphere is prevented.—H. S. H.

*Ceramic ware; Process for preheating and smoking — in ring furnaces.* G. Korngiebel. G.P. 331,677, 19.7.17.

The chambers of the preheater are separated from one another by dampers which do not reach to the bottom of the combustion flue. The smoke and gases are removed through slits in the bed of the

hearth, while the steam rises up through the chambers and is thereby separated from and prevented from saturating with moisture the cooled smoke and from flowing from heated to cooler chambers, and so being repeatedly condensed and evaporated.—A. R. P.

*Refractory insulating material; Manufacture of — from aluminium and alkali silicates and combustible fillers.* L. Kern. G.P. 330,064, 11.5.19.

The mixture of silicates and filling material is worked to a paste with a solution of aluminium hydroxide in a dilute mineral acid, and yields on calcining a product of high porosity.—L. A. C.

*Glass-pot furnace.* M. Mathy. U.S.P. 1,369,453, 22.2.21. Appl., 26.6.19.

See E.P. 135,115 of 1919; J., 1920, 25 A.

*Retorts for burning ceramic ware.* G.P. 332,131. See X.

#### IX.—BUILDING MATERIALS.

*Agglomerant; Special ferriferous — and its applications in presence of puzzuolanic materials.* F. Ferrari. Giorn. Chim. Ind. Appl., 1921, 3, 53–56. (Cf. J., 1920, 821 A; 1921, 11 A.)

PORTLAND cement in which the sesquioxides are present in the proportions of their equivalents, although exhibiting all the qualities of cements of this type, should be free from binary compounds of aluminium and iron. Making use of a mixture composed of limestone (87.6% CaCO<sub>3</sub>), clay, silicious sand, and ferric oxide obtained by burning pyrites, the author has obtained a cement of the percentage composition: SiO<sub>2</sub>, 24.04; insoluble silicious residue, 0.28; Al<sub>2</sub>O<sub>3</sub>, 3.32; Fe<sub>2</sub>O<sub>3</sub>, 5.24; CaO, 65.10; MgO, 0.47; SO<sub>3</sub>, 0.61; alkali (not determined), 0.94. Such cement is stable towards sulphates and chlorides and is to be regarded as a product of more easy scorification and of a greater content in hydraulites (calcium silicates). It is capable of replacing, in all their applications, not only iron-Portland cement, but also ordinary Portland cement. Addition to such cements of puzzuolanic substances so as to modify their high basicity, renders them suitable for giving absolutely impermeable mortars, which are of general application and are mechanically superior to those having a basis of ordinary Portland cement.—T. H. P.

*Blast-furnace slag, and its use as building material.* R. Grün. Z. angew. Chem., 1921, 34, 101–102.

The composition of blast-furnace slag varies between wide limits. It may contain 24–70% SiO<sub>2</sub>+MnO, 3–30% Al<sub>2</sub>O<sub>3</sub>+Fe<sub>2</sub>O<sub>3</sub>, and 17–60% CaO+MgO. For use as a hydraulic cement, finely ground slag is first calcined with limestone, and the resulting "Portland cement clinker" is mixed with granulated slag in the proportions of 15:85. Another composition, known as "iron Portland cement," consists of 30% of slag and 70% of the clinker. Even slags, hitherto considered worthless, have been rendered suitable by the addition of lime and alumina, and heating the mixture in an electric furnace, but there remains a wide field for further investigation.—W. J. W.

*Insulating and building materials; Relative heat conductivities of some —.* J. J. Lichtin. Chem. and Met. Eng., 1921, 24, 368.

The relative heat conductivity of "Porete" (an insulating material made from cement and sand), and of various other materials, was determined by

the "air box" method. Cubical boxes, 8x8x8 in., with walls 1 in. thick, were heated internally by an electric incandescence lamp. Temperatures were observed by means of two thermometers inserted through the walls, and three thermometer bulbs in contact with the outer surface of the box. The values for the conductivity expressed as the quantity of heat in B.Th.U. which flows through 1 sq. ft. of area, through a unit thickness of 1 ft., having a temperature difference of 1° F. between its faces, were as follows, the specific gravities of the various materials being given within brackets:—Concrete, 1 cement : 2 sand (2.01), 0.1760; gypsum board (1.00), 0.0846; "Porete" reinforced with expanded metal (0.98), 0.0786; yellow pine from N. Carolina (0.64), 0.0719; air cell asbestos board (0.21), 0.0335; cork board (0.187), 0.0277. The values apply only under similar conditions of experiment, which are approximately those under which the materials are used ordinarily as insulators or building materials.

—C. A. K.

#### PATENTS.

*Bricks, blocks, slabs, tiles, etc.; Manufacture of — for paving or building or other purposes.* C. M. C. Hughes, A. E. Brown, and T. Hill-Jones, Ltd. E.P. 159,239, 13.9.19.

Bricks etc. are prepared by grinding and mixing together clinker or slag (60 pts.), limestone dust (27 pts.), bitumen (10 pts.), making the mass plastic by the addition of creosote oil (3 pts.) and if necessary water or sodium silicate solution, and pressing the bricks etc. from the plastic mass. They are dried and then heated sufficiently to flux the bitumen, which binds the material on cooling. In order to prevent clogging of the pistons of the press, it is sometimes necessary to mix and heat the bitumen, oil, and limestone together and, after cooling, to grind this binder finely before mixing with the main body as described above.—H. S. H.

*Heat non-conducting compositions.* J. C. S. McLay. E.P. 159,411, 28.2.20.

A mixture of 75% of waste calcium carbonate from the manufacture of soap, 20% of sawdust, 5% of flour sweepings added as an agglutinating material, and 5% of ammonium carbonate is heated in the plastic condition, when the ammonium carbonate acts as an aerating agent.—H. S. H.

*Artificial [lithographic] stone.* J. Kiermayer, and Ver. Chem. Fabr. zu Leopoldsdahl, A.-G. G.P. 329,592, 26.6.19.

A stone free from air bubbles is prepared without treatment under vacuum, by mixing magnesia with an excess of magnesium chloride solution, stirring to release air bubbles, and then removing the excess solution.—L. A. C.

*Lime; Manufacture of volume-stable, dry —.* J. Joachim and J. Schulte. G.P. 329,943, 21.2.19.

QUICKLIME is slaked in open vessels by means of superheated steam, and either before or during the process is heated above 100° C.—L. A. C.

*Concrete; Process for the preparation of a — free from shrinkage cracks.* A. Guttman. G.P. 330,784, 29.1.20.

So much plaster, or other material which acts in a similar manner, is intimately mixed with the finely ground cement or cement mixture that the natural contraction of the cement on setting is counter-balanced or exceeded by the expansion due to the added material.—A. B. P.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Cast iron; Manufacture of — from burnt pyrites in the electric furnace.* M. Guédras. Giorn. Chim. Ind. Appl., 1921, 3, 58—61.

In the Guédras and Duina process, the burnt pyrites is first completely dried, then almost completely freed from sulphur, and agglomerated, in a continuous rotary furnace of the Breuillé type heated by lignite gas or blast-furnace gas; in this way the percentage of sulphur is reduced from 2—3 to 0.1—0.2, and the resulting product is hard yet highly permeable to gases. The burnt pyrites is then introduced into the fusion hearth of an electric furnace, where it undergoes reduction and further desulphurisation, the latter being facilitated by addition of dry calcium chloride:  $\text{FeS} + \text{CaCl}_2 + \text{CO} = \text{Fe} + \text{CaS} + \text{COCl}_2$ ; the carbonyl chloride liberated is rapidly decomposed, by means of moist air, into hydrogen chloride and carbon dioxide. The percentage of sulphur in the cast iron obtained is 0.01—0.03.—T. H. P.

*Steel; Dimensional limitation of successive heat-treatments of carbon —.* W. P. Wood. Chem. and Met. Eng., 1921, 24, 345—356.

UNIFORM samples, 1 cm. square and 10 cm. long, of steel bars ranging in carbon content from 0.11 to 1.14%, were measured in a micrometer, and subjected to a hardening quench in oil from 900° C., followed by a draw at 600° C., in order to bring all possible effects to a maximum value. After the first complete heat treatment the hypo-eutectoid steels were shorter than in the original annealed state, the eutectoid steels had not changed appreciably in length, and the hyper-eutectoid steels showed slight dilatation. The results in general are held to be in agreement with the view of Andrew and others (J., 1920, 452 A), according to which two opposing influences affect carbon steels which are heated slowly to 1000° C., namely, a contraction caused by the conversion from  $\alpha$ - to  $\gamma$ -iron, and a solution of carbide which causes an expansion.—C. A. K.

*Steels; Distribution of hardness in quenched carbon — and quenching cracks.* K. Honda and S. Idei. Sci. Rep. Tohoku Imp. Univ., 1920, 9, 491—507.

To study further the formation of quenching cracks in carbon steels (cf. Honda and Matsushita, J., 1919, 582 A), experiments were made to determine the distribution of hardness in quenched specimens in the form of small cubes or cylinders, by means of Shore's scleroscope. With a soft quenching, for example in oil from a temperature not exceeding 820° C., the hardness decreases from the periphery to the centre. With moderately hard quenching, for example a 0.91% C steel in water from 780° C. or a 1.47% C steel in oil from 900° C., the hardness of the specimen is uniform. A hard-quenched steel, for instance a 0.68% C steel quenched in water from 800° C. or above, is hardest towards the centre. This is explained by the presence of arrested austenite in martensite (cf. Honda, J., 1919, 821 A). The quenching cracks in small pieces occur when the hardness of the central portion is greater than that of the periphery, the cause being the stresses occasioned by the difference between the specific volumes of austenite and martensite. Since this difference increases as the temperature falls, the cracking usually takes place as the specimen approaches room temperature. The hardness of a hard-quenched specimen gradually increases with lapse of time owing to gradual transformation of the arrested austenite into martensite. To avoid cracking, quenching should be only medium hard. The temperature from which to quench a particular

steel can be determined by quenching specimens from, say, 800°, 900°, and 1000° C. in oil and determining the hardness at the centre and periphery. Hardness-quenching temperature curves are drawn for the centre and periphery, and the point of intersection of these will give the temperature from which to quench to obtain uniform hardness throughout the specimen.—E. H. R.

*Steel and iron; Mechanism of increased corrosion of — due to small copper contents.* D. M. Buck. Trans. Amer. Electro-Chem. Soc., 1921, 47-54. [Advance copy.]

PRELIMINARY results of a four-years' test on the rate of corrosion of copper steels are given after atmospheric exposure of specimens for 32 months. The specimens contained from 0.01 to 0.242% Cu, and the amount of rusting was estimated by determining the quantities of the loose film removable by brushing with a wire brush, and the closely adherent film removed later by an ammoniacal solution of ammonium citrate. A steel containing 0.1% Cu showed a loss of 3.62 oz. per sq. ft. due to loosely adherent rust, and of 6.43 oz. due to closely adherent rust, which was the minimum total loss of the series. Present data indicate that the harmful influence of sulphur on corrosion is in some manner controlled by the addition of copper, and also that the more adherent nature of the rust film formed is related to the increased resistance peculiar to copper steels.—C. A. K.

*Corrosion of old iron.* E. A. and L. T. Richardson. Trans. Amer. Electro-Chem. Soc., 1921, 55-59. [Advance copy.]

ANALYSES of fifteen specimens of iron and steel which had resisted atmospheric corrosion for upwards of 30 years show that steels which are rust-resisting contain copper. Steels containing more than 0.1% Cu were relatively in good condition; those containing less than 0.1% Cu gave contradictory evidence. A comparative test of exposure for two years showed that the older irons are no less subject to corrosion under similar conditions than recently manufactured wrought iron.—C. A. K.

*Iron and steel; Preventing corrosion of — when not exposed directly to the atmosphere.* F. N. Speller. Trans. Amer. Electro-Chem. Soc., 1921, 37-46. [Advance copy.]

EXPERIMENTS and observation in practical working have shown that the extent of corrosion of iron or steel in contact with water is directly proportional to the quantity of oxygen in solution, and that the composition of the iron is of minor account. Dissolved oxygen has been removed economically by mechanical de-aeration of the water. For practical protection of water pipes from internal corrosion at 80° C. it is not necessary to have the oxygen less than 0.4 c.c. per l., but for steam boiler work a limit of 0.2 c.c. per l. is advisable. This limit may be reached by spraying the water into a vacuum chamber, or by passing the water through a suitably designed preheater. A second method is based on fixing the free oxygen by chemical combination and has been employed in practice by passing the heated water through a storage tank packed with a mass of expanded iron giving about 60-70 sq. ft. of surface per cu. ft. of space. In 30 mins. at 77° C. the oxygen content may be reduced to 0.3 c.c. per l. Free carbon dioxide, or bicarbonate, is always increased slightly by this treatment, but practically no corrosion is found to occur in the absence of oxygen. Other metals, e.g. zinc, may be used but are more costly. A series of experiments shows that the corrosion of zinc and brass is retarded greatly by the removal of oxygen from water, and also that the accelerating effect of

copper and brass in contact with steel is reduced by the lack of a depolariser. The rate of corrosion may be retarded by increasing the proportion of hydroxyl ions in relation to the hydrogen ions present in the water, as by addition of lime or soda ash, but the formation of an insulating scale may cause other difficulties. The results of recent research on the corrosion of hot water feed service piping are tabulated.—C. A. K.

*Ferro-silicon; Influence of silicon upon the properties of —.* A. T. Lowzow. Tidskr. Kemi, 1919, 16, [1]. Chem. and Met. Eng., 1921, 24, 481-484.

A SERIES of commercial alloys containing from 49.1% to 93.41% Si were examined by microscopical and chemical means, and from the results obtained the equilibrium diagram was constructed. The compound FeSi forms an eutectic with silicon at 55% Si and 45% Fe. The compound FeSi<sub>2</sub> is a decomposition product formed from the eutectic by slow cooling. Determinations of the specific gravities of the various alloys indicate that a fairly accurate estimation of the silicon content can be quickly obtained by determining the specific gravity, although slight variations are produced by the introduction of impurities.—J. W. D.

*Ferronickels; Effect of additions on the anomaly of dilatation of —: application to iron-nickel-chromium alloys.* P. Chevenard. Comptes rend., 1921, 172, 594-596.

THE curve showing the variation of the total anomaly of dilatation,  $A_t$ , with nickel content in the case of pure ferronickels, rises rapidly from zero at 25% Ni to reach a sharp maximum at a point corresponding to the compound Fe<sub>3</sub>Ni, and then descends, crossing the zero line at 58% Ni, reaching a minimum, and then recrossing at 80% Ni, and finally recrossing at 99% Ni after a second slight maximum. By the addition of increasing amounts of chromium similar curves are obtained, but the maximum value of  $A_t$  becomes less and less, and the maximum point is displaced more and more towards a higher nickel content. The maximum points are practically on a line which, starting from Fe<sub>3</sub>Ni, tends towards a point on the line NiCr.—W. G.

*Iron pyrites; Heat of dissociation of —.* H. Kamura. Chem. and Met. Eng., 1921, 24, 437.

IRON pyrites was decomposed at different temperatures ranging from 400° to 700° C., the decomposition pressure at which the sulphur vapour was given off being measured. From the decomposition pressure curve the heat absorbed in the decomposition was deduced thermodynamically, giving,  $Q$ , the heat of decomposition per molecular volume of sulphur vapour formed as 37,223 cal., corresponding to the heat value of the equation  $2\text{FeS}_2 = 2\text{FeS} + \text{S}_2$ . The value of the heat absorbed per atom of sulphur, or conversely the heat evolved in the combination, can therefore be expressed thermochemically as  $(\text{FeS}, \text{S gas}) = 18,611 \text{ cal.}$ —J. W. D.

*Gold ore; Treatment of antimonial — at the Globe and Phoenix Gold Mine, S. Rhodesia.* V. E. Robinson. J. Chem. Met. Min. Soc. S. Afr., 1921, 21, 117-119.

THE greater part of the coarse stibnite present in the ore is separated by hand picking, and the remainder of the ore is wet crushed by stamps, the pulp from which passes over amalgamated plates to a series of grinding pans containing mercury. The pan discharge passes over canvas strakes, then through a series of mercury traps, and finally into a classifier to separate the sands and slimes, which are stored separately and allowed to weather for some time, after which each is cyanided separately.



The concentrates from the strakes are reconcentrated and amalgamated in a pebble mill. The recoveries are 65–70% by amalgamation, and about 80% of the total gold in the tailings is recovered by cyaniding. The amalgam and the precipitate from the sand plant both contain small amounts of antimony, which is removed from the crude bullion by melting it under an oxidising flux, skimming, and then directing a blast of dry compressed air on the surface of the molten metal until no further fumes are evolved. The bullion obtained from the slimes contains a large amount of copper; it is granulated, mixed with sulphur and slowly heated in a graphite pot, when most of the base metals and some silver enter the matte and a slightly antimonial bullion is obtained, which is further purified as described above.—A. R. P.

*De-aeration of water or [cyanide] solutions; Chemical methods of —.* H. A. White. J. Chem. Met. Soc. S. Afr., 1920, 21, 105–111

For removing oxygen from cyanide solutions before precipitation of gold, and from boiler-feed water, comparative tests were made with a ferrous salt, a manganous salt, and tannin. The sample of water or cyanide solution was treated with the calculated quantity of chemical, together with lime, the mixture shaken, and allowed to stand until next day. The clear liquid was siphoned off and analysed, and the result compared with an analysis of the original sample. With tannin the dissolved oxygen was removed, and there was no reaction with the cyanides present. With ferrous ammonium sulphate all the oxygen was removed, but there was a loss of half the cyanide. Manganese sulphate removed only 77% of the oxygen; there was no loss of cyanide. Wattle bark and dried extract of the bark were also efficient, but required an alkalinity of at least 0.008% of soda. Wattle bark extract was the cheapest of the compounds tested. A few experiments were made on the effect of dissolved oxygen in boiler water on the corrosion of iron. In some cases the oxygen was partly removed with wattle extract and by passing hydrogen through, and in these cases the amount of iron dissolved, when free in the water or in contact with carbon, was very much less than in the untreated water.—J. H. J.

*Gold; Quantitative determination of — by cupellation, and the examination of large quantities of gold destined for the manufacture of coinage.* J. W. A. H. Smit. Rec. Trav. Chim., 1921, 40, 119–152.

In the cupellation of gold bullion certain details must be strictly adhered to if accurate results are to be obtained. Uniformity of access of air in the furnace is necessary and too rapid a cupellation is injurious. The surface and porosity of the cupel are not of such importance as its thermal conductivity. The ratio of lead to copper must be at least 55:1 and may be higher. Heating of the button after iridescence is necessary for commercial samples of gold, and in this case the best ratio of silver to gold is 2:1. The cornets should be well heated both before and after boiling with acid, and it is advisable to roll the buttons into sheets 0.15 mm. thick. The nitric acid used for the extraction must be absolutely free from chlorine and of a somewhat higher concentration (36° B., sp. gr. 1.33) than that generally used.—W. G.

*Zinc dust and scrap; Electro-metallurgical process for working up — to the pure metal.* H. Paweck. Z. Elektrochem., 1921, 27, 16–21.

In working up brass for copper by an oxidation process large quantities of zinc dust are obtained. This dust is worked up as follows: The mass is treated with a slight excess of dilute sulphuric acid,

filtered, the solution treated with zinc scrap to precipitate copper, filtered, and treated with the calculated quantity of bleaching powder to oxidise and precipitate iron. The solution is then used to make the anolyte and catholyte for the subsequent electrolysis. The electrolysis is carried out in a two-diaphragm cell, the cathode part being between the two anode parts and between the two diaphragms. The anodes are lead plates, 3.35 sq. dm. surface, and the cathode an iron plate. The anolyte contains 4% of zinc and 5% of free sulphuric acid, whilst the catholyte is neutral and contains 10% of zinc. The electrolysis is carried out with a current of about 4.1 amps. and 5.5 initial voltage which falls to 3.65 volts in 19 hrs. The process is stopped when the zinc concentration has fallen to 4%; at the same time the acid content of the catholyte has risen to 5% and that of the anolyte to 10%. The catholyte is used in the next operation as anolyte and the original anolyte is treated with more zinc dust and used as catholyte. By this process 1 kg. of zinc is obtained for the expenditure of 3.38 k.w.-hrs.; this corresponds to a current yield of 99.08%. A similar process for working up the worn out zincs of batteries is described; in this the zincs are placed in the anode cell and used as the anode.—J. F. S.

*Lead; Electrolytic corrosion of — by continuous and periodic currents.* E. R. Shepard. Trans. Amer. Electro-Chem. Soc., 1921, 23–36. [Advanco copy.]

THE rate of corrosion of lead submerged under water and in moist earth was determined under the influence of continuous and periodically reversed currents, with a view to amplify the work of McCollum and Ahlborn (U.S. Bur. of Standards, Tech. paper No. 72). The results agree generally with those of the earlier work, except that a coefficient of corrosion of 100% (equal to the theoretical value) may be obtained in earth with very low direct current densities (0.93 amp. per sq. ft.). The earth must be saturated with water and pressed firmly around the specimen in order to obtain this value. The time factor does not apparently influence the coefficient of corrosion of lead in earth, but in water a reduction occurs from 100% after 20 hrs. to 65% at 90 hrs. With periodically reversed currents having an algebraic average of zero, the coefficient, calculated on the basis of anodic current, decreases rapidly in earth, reaching a value of 14% after 465 hrs. Under alternating conditions the low coefficients of corrosion obtained are due probably to a re-deposition of lead during the cathodic period, or an equivalent reduction process at the cathode. When the positive and negative values are unequal, the coefficient increases with the percentage of the total ampere-hrs. under anodic conditions.—C. A. K.

*Nickel.* W. von Selve. Z. Metallk., 1921, 13, 40–46.

A GENERAL account of the degree of purity and physical properties of the commercial forms of nickel, and a brief outline of the technical applications of the metal.—E. H. R.

*Chromium; Cause of periodic phenomena in the electrolysis of chromic acid and the deposition of metallic —.* E. Liebreich. Z. Elektrochem., 1921, 27, 94–110.

WHEN a solution of chromic acid is electrolysed, using iron, chromium, or platinum electrodes, regular periodic, sudden variations of both the P.D. and of the current are sometimes observed, in such a manner that as the P.D. increases the current falls slightly, and vice versa. A study of the P.D.-current density curves of a chromic acid cell over a range of P.D. from 1 to 7 volts, using a 22% solution of chromic acid and platinum electrodes,



showed three breaks in the curve, and, in effect, it is shown that the chromic acid solution has four characteristic P.D.-current density curves above 1.7 volts, all approximately parallel. Each curve corresponds with a specific electrolytic chemical reaction. Over certain ranges of current density two or more of these reactions are possible, each requiring a specific P.D., and the periodic variations of P.D. and current observed are due to the periodic sudden change of the course of the electrolysis from one reaction to another. On the first two curves, no hydrogen is evolved from the cathode but a slight deposit is formed of chromi-chromate. On the third curve there is a slight cathodic deposit of a different kind and some evolution of hydrogen, whilst on the fourth curve there is vigorous evolution of hydrogen and metallic chromium is deposited on the cathode. The chemical reactions characteristic of the four curves are probably of the types: (1)  $\text{Cr}^{VI} + \text{Cr}^{III}$ ; (2)  $2\text{CrO}_3 + 6\text{H}^+ \rightarrow \text{Cr}_2\text{O}_3 + 3\text{H}_2\text{O}$ ; (3)  $\text{Cr}_2\text{O}_3 + 2\text{H}^+ \rightarrow 2\text{CrO} + \text{H}_2\text{O}$ ; (4)  $2\text{H}^+ + \text{H}_2$ , and  $\text{CrO} + 2\text{H}^+ \rightarrow \text{Cr} + \text{H}_2\text{O}$ . When iron or chromium cathodes are used, metal passes into solution except on curve (4). Generally deposition of metallic chromium only occurs with a current density of at least 0.5 amp. per sq. cm., but under certain conditions solutions can be obtained which give a chromium deposit at a much lower current density, for instance by heating chromic acid crystals below the incandescent point so as to cause them to dissolve in their water of crystallisation and then diluting with water. Such a solution has an abnormally low conductivity and gives a firmly adhering, brilliant deposit of metallic chromium, instead of the usual grey, loosely adhering deposit. A similar result was obtained by dissolving in water and electrolysis the chromo-chromate deposit obtained from the electrolysis characteristic of curve (3) above. Alkali chromite solutions, obtained by adding potassium hydroxide to a solution of chrome alum, also deposit adherent metallic chromium at a low current density.

—E. H. R.

*Chromite; Iodometric method for determining chromium in* —. E. Little and J. Costa. J. Ind. Eng. Chem., 1921, 13, 228–230.

A mixture of 0.4 g. of chromite and 5 g. of sodium peroxide is heated in an iron crucible for 5 mins., then fused for 15 mins., cooled, and dissolved in 150 c.c. of water; the solution is treated with a further 0.5 g. of peroxide, boiled to destroy the excess of the latter, cooled, and hydrochloric acid added until all ferric hydroxide has dissolved. 5 c.c. of concentrated hydrochloric acid is then added for each 100 c.c. of solution, an excess of ammonium fluoride is introduced, the mixture is treated with 3 g. of potassium iodide and, after 3 mins., the liberated iodine is titrated with standard thio-sulphate solution.—W. P. S.

*Blast-furnace slag.* Grün. See IX.

*Tungsten.* Fiorentino. See XXIII.

## PATENTS.

*Ferro-chrome alloys; Production of* —. W. B. Ballantine. E.P. 159,280, 15.11 and 5.12.19.

The special alloy constituents, e.g., tungsten and vanadium, are added to the steel bath as ferro-alloys in compacted form either as briquettes or in canisters, and contain in their compact mass a thermo-reducing mixture made up of a compound of a metal desired in the alloy, e.g., chromite, and a thermo-reducing agent such as aluminium. The thermo-reducing mixture is added in such proportion that on ignition in the molten bath the mixture liberates sufficient heat to maintain the bath at the temperature necessary to ensure complete reaction of all the constituents.—J. W. D.

*Magnetic or weakly magnetic iron ore containing magnetic ferrous sulphide as a secondary constituent; Process and apparatus for treating* —. R. Stören and R. Johanson. E.P. 159,380, 18.12.19.

This ore is subjected to a reducing treatment with hydrogen at 400°–500° C. in a continuously working furnace, whereby the oxide content is partially reduced while the sulphide constituents are rendered permanently non-magnetic. The material leaving the reducing furnace is suspended in water and subjected to magnetic separation, the separated oxide being conveyed to a second reducing furnace and reduced to metal, which is transferred to an electric melting furnace.—J. W. D.

*Ferrosilicon-aluminium; Production of* —. W. B. D. Penniman and R. S. White. U.S.P. 1,369,298, 22.2.21. Appl., 9.6.19.

Compounds of aluminium, silicon, and iron are treated with a reducing agent in an electric furnace whereby a ferro-silicon-aluminium alloy is produced.—J. W. D.

*Steel; Non-magnetic forgeable* —. F. Krupp, A.-G. G.P. 298,429, 13.11.15.

The steel contains 10–15% Ni, 6–4% Mn, and 5–2% Cr. The chromium may be replaced partly or completely by tungsten, molybdenum, or vanadium.—L. A. C.

*Iron; Separation of other metals from* —. Koelsch. G.P. 330,131, 7.5.14.

Iron may be separated from other metals, e.g., stripped of a plating of copper or nickel, by treating the mixed metals with a solution containing an excess of nitrite ions and a low concentration of hydrogen ions. The process, however, is not applicable to the detinning of iron.—A. R. P.

*Silico-ferromanganese or silicospiegel; Production of* — in the blast furnace. C. Stöckmann, sen. G.P. 330,196, 25.2.19.

In order to minimise the loss of manganese when making silicospiegel the charge is so made up that the oxygen content of the bases ( $\text{Al}_2\text{O}_3 + \text{CaO} + \text{MgO}$ ) in the resulting slag is as great as, or greater than, that of the silica, by the addition, if necessary, of sand or quartz to the charge, which addition also serves to reduce the alumina content of the slag if that is too high.—A. R. P.

*Zinc and iron; Recovery of* — from zinciferous iron ores by smelting in the blast furnace. F. Burgers. G.P. 330,396, 8.5.19.

Zinciferous iron ores and roasted sulphide products containing up to 50% Zn are smelted in a metal-jacketed blast furnace lined with carbonaceous bricks, whereby the zinc is volatilised and subsequently recovered from the issuing gases.—A. R. P.

*Metals [auctile tungsten] and their manufacture.* British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 155,951, 17.4.17.

A mixture of 1400 g. of commercial tungstic acid, 620 g. of sodium carbonate, 620 g. of potassium carbonate, and 300 g. of sodium nitrate is made into a paste with water, dried, and fused. The melt is dissolved in water, the solution filtered and diluted to 6 l., giving solution A. A mixture of 180 g. of commercial silicic acid, 620 g. of potassium carbonate, and 150 g. of sodium nitrate treated in a similar manner gives solution B. 250 c.c. of B is added to 3 l. of A, the mixture boiled and poured into 3 l. of a boiling solution of hydrochloric acid (1:1). The precipitate is filtered off, washed with water till the washings come through turbid, dried, ignited, and reduced by hydrogen in 160-g. lots.

This metal ("X metal") can be pressed, sintered, and drawn into wire, but better results are obtained by grinding equal weights of it and the oxide from which it was produced and reducing the mixture in hydrogen in 150-g. lots. In the above process the silica may be replaced by tantalum or columbium pentoxides. An alternative process is to reduce the residue obtained by evaporating tungstic acid with boric acid solution or, preferably, to mix 1 kg. of the tungstic acid precipitate produced in the first process with 12.5 g. of boric acid in 500 c.c. of water, dry, ignite, and reduce the residue. The powder produced by the last process ("Z metal") is blacker and more bulky than that produced by the former processes. In all cases the metal obtained is very pure, of a fine-grained structure, and when drawn into wire does not sag at high temperatures. Filaments prepared from the wire are given a preliminary heat treatment by running them at 30 volts for 2 min., then at steps of 5 volts for 2 min. up to 60 volts, finally at 15% over-voltage for 15 mins.—A. R. P.

*Tungsten ores; Process and apparatus for the treatment of —.* J. C. Blanch. G.P. 330,987, 28.3.20.

THE ore, after decomposition with alkali chloride and, if necessary, nitrate, is roasted, and the resulting mass is treated with dilute sulphuric acid in a vessel in the form of an inverted conical funnel through which steam is passed in such a manner that the solids are vigorously agitated. The resulting solution of the bases in the ore with tungstic acid in suspension is delivered by tilting the funnel-shaped vessel into a series of decanting vessels in cascade, in which vessels the tungstic acid is recovered by settling and decantation, and is subsequently reduced to metal.—A. R. P.

*Tin; Recovery of — from tin-pot skimmings, with zinc chloride as a by-product.* A. J. and H. D. Evans. E.P. 158,926, 23.9.19.

TIN-POT skimmings are ground with a slag-forming mixture (glass 3 pts., felspar 1, fluor spar 1 pt.) to a thin paste containing flattened pieces of tin. After draining and washing on a filter cloth the liquor may be evaporated for the recovery of zinc chloride and the residue smelted with addition of soda ash. The slag produced during smelting may be used in the preliminary grinding process.—C. A. K.

*Tin; Winning of —.* J. J. Collins. E.P. 159,071, 10.12.19.

TIN-BEARING rock is crushed, treated with reducing gases to convert tin oxide into metallic tin, and then subjected to the action of chlorine gas, preferably obtained by the expansion of anhydrous liquid chlorine. The stannic chloride formed is evaporated or extracted by means of solvents, and metallic tin is recovered by replacement of the tin by zinc.—C. A. K.

*Aluminium alloys.* J. E. Hurst and E. B. Ball. E.P. 159,008, 18.11.19 and 5.3.20.

LIGHT aluminium alloys containing 0.5–6% Cr, and not more than 6% of additional elements, such as Cu, Ni, Zn, Mg, Sb, are suitable for castings which are subjected to high temperatures, e.g., parts of internal combustion engines. It is preferred that the total percentage of additional elements does not exceed the chromium content.—C. A. K.

*Ores and the like; Treatment of — by flotation.* M. T. Taylor and J. W. Partington. E.P. 159,025, 21.11.19.

In the flotation treatment of ores, particularly for the separation of tin and tungsten, the flotation

agent may be anionated fatty or resin acid, which may be dissolved in a fatty acid ester, e.g., propyl, butyl, or amyl acetate, or in a cyclic compound containing nitrogen, e.g., pyridine. Phenolic substances, or other oils used in flotation practice, may also be added.—C. A. K.

*Metallic surfaces; [Electrolytic] cleaning of —.* T. A. Edison. U.S.P. 1,369,271, 22.2.21. Appl., 3.7.19.

METALS are cleaned in an electrolytic cell using as an electrolyte a solution of an alkali sulphate and sodium or potassium hydroxide.—J. W. D.

*Cinnabar ore; Apparatus for treating —.* A. J. Garver. U.S.P. 1,369,738, 22.2.21. Appl., 26.12.19.

THE apparatus comprises a rotary condenser, a device for directing gas and fumes from a retort into the condenser, and a water-discharge pipe communicating with this device.—J. W. D.

*Manganese-copper alloys; Ferruginous —.* Iaa-bellenhütte, G.m.b.H. G.P. 303,864, 4.2.17.

MANGANESE-COPPER alloys containing also, if required, other metals, such as zinc, tin, or aluminium, are treated with a certain amount of silicon, preferably as ferro-silicon. The resulting alloy, when molten, runs more freely, and on cooling no segregation of constituents rich in iron occurs.—A. R. P.

*Metals; Process and furnace for recovering — from their ores by smelting.* H. Dahlem. G.P. 330,290, 13.6.19.

HEAT is supplied to the furnace by passing a mixture of compressed air and gas through a series of tuyères situated one above the other, so that each zone of the furnace can be independently heated to the required temperature. Means are provided for regulating the air supply so as to obtain the maximum utilisation of the heat.—A. R. P.

*Metals and alloys; Process for coating substances having a large surface with —.* Chem. Fabr. von Heyden A.-G. G.P. 330,306, 21.5.20.

THE metal or alloy is disintegrated by an electrical method in a suspension in a suitable liquid, e.g., water, alcohol, ether, or oils, of the substance to be coated, if necessary, after the addition of a reducing agent. For example, nickel is disintegrated under a 1% aqueous solution of hydrazine hydrate containing bole in suspension.—A. R. P.

*Metallic oxides; Reduction of — by means of sodium alloys.* W. Schuen, H. C. Grosspeter, and A. Kemper. G.P. 330,679, 20.2.17.

METALLIC oxides, e.g., those of manganese or chromium, are reduced to the corresponding metal by heating them with finely divided alloys of sodium with one or more of the metals—iron, manganese, or silicon, preferably NaSi, on the hearth of a Siemens-Martin furnace or in other suitable apparatus. These sodium alloys may also be used for deoxidising molten metals such as iron or steel.—A. R. P.

*Regenerative furnace for refining copper, brass, and other alloys.* F. Weeren. G.P. 307,704, 31.8.17.

THE hearth of the furnace is connected by means of two different interchangeable heads, on each side of the furnace, with two independent chamber systems, in one of which the air is preheated and in the other the gas. This process avoids the difficulty experienced in carrying out the refining in regenerative furnaces of the ordinary type, due to large quantities of oxides being deposited in contact with the chequerwork of the regenerators.—A. R. P.

*Roasting furnace for pyrites, spent oxide, or the like; Rotary* —. Rocholl. G.P. 332,130, 27.7.19. Addn. to 326,441 (J., 1921, 183 A).

THE interior of the front part of the cells is made conical, and the discharge openings for the roasting gases are arranged in an axial direction from these conical ends. This arrangement allows the charge to pass rapidly from the radial charging channels into the roasting chambers and prevents it from falling back into the channels during rotation. The gases also are still more readily removed from the apparatus than by the previous arrangement.

—A. R. P.

*Metals; Process for rendering the surfaces of — more highly resistant to attack.* R. Walter. G.P. 330,707, 11.3.17.

THE metal to be treated is coated with a relatively thin layer of a more highly resistant metal in the liquid state, and welding of the two metals is effected by electrical or thermal methods. As coating metals, alloys of iron or chromium, or of metals of the iron and chromium groups with copper and, preferably, metals such as vanadium or titanium, used in alloy steels, are suitable, e.g., alloys consisting of 60% Fe, 30% Cr, and 10% Ni, or of 70% Fe, 15% W, 10% Ni, and 5% Cu, have a high mechanical strength and resistance to oxidation.—A. R. P.

*Copper, zinc, and silver; Process for sulphatising sulphide ores and products containing —. Process for producing metallic oxides and sulphur dioxide from solutions of metallic sulphates.* W. Buddeus. G.P. (A) 331,176, 27.11.19, and (B) 331,177, 12.11.19.

(A) THE finely powdered ore is mixed with solutions of salts of very difficultly fusible oxides, and the mixture, after being made into briquettes and dried in a shaft furnace, is roasted. In this way sintering of the charge is avoided, and owing to the porous nature of the charge the air is enabled to come into contact with every particle of it, thus preventing "kernel" roasting. (B) Concentrated solutions of zinc sulphate, or of other metallic sulphates, such as those of magnesium or copper, are mixed with a finely divided carbonaceous fuel and the mixture is rapidly dried in thin layers on rolls. The dried mixture is smelted, after the addition of more fuel, if necessary, in a blast furnace or converter by means of compressed air, or it may be heated in a retort or other suitable furnace after briquetting. The sulphur dioxide evolved is used for making sulphuric acid, while the oxide obtained in the case of zinc is very dense and suitable for use directly in the distillation process as it is practically free from sulphur.—A. R. P.

*Zinc; Diagonal retorts for the distillation of — or for the burning of ceramic ware or the like.* H. V. Dahlem. G.P. 332,131, 12.7.19.

THE material to be treated is prevented from coming in contact with the walls of the retort by enclosing it in a specially constructed receptacle which fits into the retort and can be readily replaced when the operation is finished. These receptacles are rounded on the front side to prevent their sticking in the retorts, and are provided with a channel on the under-side, so that by pushing a rod under them they can easily be lifted up in case of accident.

—A. R. P.

*Iron and alloys thereof; Process of electrolytically producing —.* A. T. C. Estelle. E.P. 159,906, 27.3.18.

SEE U.S.P. 1,275,161 of 1918; J., 1918, 659 A.

*Minerals or products containing iron and titanium and vanadium; Treatment of —.* B. p. F. Kjelberg. E.P. 159,532, 20.10.19.

SEE G.P. 324,581 of 1919; J., 1920, 783 A.

*Roasting furnaces.* A. Zavelberg. E.P. 2690, 2.2.14. SEE F.P. 463,685 of 1914; J., 1914, 970.

*[Metallurgical furnaces;] Heat regenerator [for —].* L. B. Skinner. Reissue 15,052, 22.2.21, of U.S.P. 1,350,267, 17.8.20. Appl., 15.12.20. SEE J., 1920, 661 A.

*Aluminium; Process for increasing the resistance of — to acid and alkaline liquids.* Metallindustrie Schiele und Buchsaler. E.P. 140,069, 6.3.20. Conv., 8.3.19.

SEE G.P. 318,141 of 1919; J., 1920, 415 A.

*Metals; Surface treatment of —.* British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 159,102, 19.3.20.

SEE U.S.P. 1,365,449 of 1921; J., 1921, 152 A.

*Separating soluble from insoluble matter [tungstic acid]; Apparatus for —.* J. C. Blanch. E.P. 160,081, 15.3.20.

SEE G.P. 330,987; preceding.

*Fusible materials [metals]; Nozzles for disintegrating —.* E. J. Hall. E.P. 140,794, 25.3.20. Conv., 25.3.19.

*Drying material.* E.P. 139,478. See I.

*Blast-furnace gas.* E.P. 148,802. See I.

*Pickling solutions.* U.S.P. 1,369,451. See VII.

## XI.—ELECTRO-CHEMISTRY.

See also pages (A) 253, *Electrolytic reactions of naphthalene* (Ono). 257, *Reaction  $3\text{HNO}_3 \rightarrow 2\text{NO} + \text{HNO}_2 + \text{H}_2\text{O}$*  (Klemenc); *Potassium perchlorate* (Blau and Weingand). 258, *Cathodic reduction of nitrogen* (Tiede and Schleede). 263, *Working up zinc dust and scrap* (Paweck); *Electrolysis of chromic acid* (Liebreich); *Corrosion of lead* (Shepard). 282, *Separating copper, tin, and antimony* (Foerster and Aanensen).

### PATENTS.

*Insulating and protecting electrical apparatus; Compositions for — and for other purposes.* The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 159,421, 17.3.20.

A COMPOSITION which can be baked at relatively high temperatures without blistering, is made by dissolving a resin, such as shellac, copal, or rosin, in an aqueous solution of ammonia and mixing with an alkali silicate. A filler, such as siliceous, and a colouring matter, such as ultramarine blue, may be added.

—J. S. G. T.

*Electric furnace.* M. R. Trembour, Assr. to Ludlum Electric Furnace Corp. U.S.P. 1,369,578, 22.2.21. Appl., 1.10.19.

AN electric furnace comprises a hearth upon which the charge rests, top electrodes some of which are maintained in arcing relation to the charge during operation of the furnace, and an electrical connexion to the marginal area of the hearth.

—J. S. G. T.

*Fixation of gases; Electric —.* G. T. Southgate. U.S.P. 1,369,714, 22.2.21. Appl., 11.4.18.

THE gases are delivered through porous electrodes arranged within the reaction chamber and connected with conductors extending outside the chamber. Gas induction tubes are connected with

the electrodes, and the reaction chamber is provided with a tube for conveying away the products of reaction.—J. S. G. T.

*Electric flaming arc; Process and apparatus for treating gases in the —.* E. Kneip. G.P. 330,079, 7.9.19.

THE electrodes between which the arc is struck are in the form of endless bands, cables, or chains, flexible tubes, etc., of metal, alloy, or other conducting material. The electrodes are supported upon conductors in such manner that the distance between them increases after the manner of the horns of a lightning arrester.—J. S. G. T.

*Electro-osmosis; Diaphragm for —.* A. Nathansohn. G.P. 329,720, 12.10.19.

THE diaphragms are composed of metallic peroxides, such as lead dioxide, manganese dioxide, either alone or mixed with binding material. Such diaphragms are suitable for use in the electro-osmosis of mixtures of albumin and ammonium sulphate, e.g., serum preparations.—J. S. G. T.

*Electrodes composed of carbon and powdered pyro-lusite; Compressed positive — for galvanic cells.* F. Christ. G.P. 329,728, 4.7.19.

THE texture of the electrodes is so adjusted either by means of external pressure or by choice of the degree of granulation of the powder, that the size of the pores in the electrodes increases gradually towards the interior, whereby the deposition of deleterious particles, e.g., crystals of ammonium salts, upon the surface of the electrodes is prevented, owing to the velocity of diffusion being greater at the surface of the electrode than towards the interior thereof.—J. S. G. T.

*Storage battery; Electric —.* H. Bardt. G.P. 329,787, 19.11.19.

A SOLUTION of a perchlorate is used as electrolyte, in conjunction with electrodes of indifferent metals. Thus a solution of lead perchlorate or copper perchlorate may be used, and the positive electrode, disposed horizontally at the bottom of the cell, is covered with the amount of lead oxide corresponding with the amount of metal deposited from the electrolyte during the charging process, which converts the lead oxide into lead peroxide.

—J. S. G. T.

*Electrodes of large cross-sectional area; Production of —.* Ges. für Teerverwertung m.b.H. G.P. 329,904, 28.11.19.

A NUMBER of smaller electrodes are combined so as to form one electrode of the desired cross-sectional area, and this is consolidated by baking in a furnace.—J. S. G. T.

*Dry cell.* A. W. Schorger, Assr. to Burgess Battery Co. U.S.P. 1,370,052, 1.3.21. Appl., 25.1.19.

SEE E.P. 135,502 of 1919; J., 1920, 824 A.

*Galvanic battery with unalterable electrolyte; Primary —.* E. W. Jungner. U.S.P. 1,370,119, 1.3.21. Appl., 16.8.18

SEE E.P. 118,843 of 1918; J., 1920, 373 A.

See also pages (A) 247, *Electrical treatment of gases* (E.P. 158,982); *Electrodes* (G.P. 331,381), 248, *Electrodes* (331,590); *Electrical purification of gases* (G.P. 332,110), 249, *Liquefaction of gases* (G.P. 329,361), 258, *Chromous chloride* (U.S.P. 1,369,204), 265, *Cleaning metals* (U.S.P. 1,369,271), 275, *Extracting foodstuffs* (G.P. 329,505).

## XII.—FATS; OILS; WAXES.

*Oil-bearing and textile plants; Development of albumin and oil in the seeds of —.* Kleberger. Chem. Umschau, 1921, 28, 2—5.

THE albumin and oil content of the seeds of rape, poppy, flax, hemp, and cameline (German sesame) were determined in the green, yellow (fully grown), and fully ripened stages, the experiments being carried over a period of four years. The highest content of nitrogenous substances was found in the green stage, diminishing in the subsequent stages of ripening. In the yellow stage the nitrogen is mainly present in the non-albuminoid condition and as amides, etc., true albuminoids, however, preponderating in the fully ripe stage. In the green stage, the "fatty substances" consist chiefly of resins and waxy substances, and exist in relatively small proportion. As the seeds develop to the fully ripe stage, the proportion of true fats increases at the expense of the resins and waxes. Hempseed shows an exceptional behaviour in being nearly at its full state of development of albumin and fat in its yellow-ripe stage. From experiments carried out by storing seeds collected in their different stages of ripeness for a period of two months, it was shown that the maximum yield of oil and albumin is obtained by harvesting the seeds in the fully ripe stage, loss of water resulting without any other corresponding changes taking place.—A. de W.

*Herring oil; Composition of —.* C. Grimme. Chem. Umschau, 1921, 28, 17—19.

THE proximate analysis of a sample of herring oil fatty acids gave the following figures: saturated fatty acids, 20%; oleic acid, 20%; linolic acid, 33%; linolenic acid, 17%; clupanodonic acid, 9%. The saturated fatty acids were determined by the lead salt-ether method, whilst clupanodonic acid was separated as octobromide from the accompanying linolenic acid hexabromide precipitated in an ether and acetic acid solution, by extraction of the mixed bromides with hot benzene. Linolic acid tetrabromide was separated from oleic acid dibromide by solution of the latter with petroleum ether of low boiling-point from the residue left on evaporation of the filtrate from the octo- and hexabromides. The author does not consider the existence of Tsujimoto's highly unsaturated acid,  $C_{22}H_{34}O$  (J., 1920, 825 A), to be proved.—A. de W.

*Otoba butter; An analysis of —.* W. F. Baughman, G. S. Jamieson, and D. H. Brauns. J. Amer. Chem. Soc., 1921, 43, 199—204.

A SAMPLE of otoba butter from the fruit of *Myristica otoba*, had sp. gr. 0.9293 at 20°/20° C.;  $n_D^{20}$  = 1.4710; iodine value (Hanus), 54.0; saponif. value, 185.0; m.p., 34.0° C., and contained 9.3% of essential oil; unsaponifiable matter (other than essential oil), 20.4%; fatty acids, 67.6%. The essential oil consisted chiefly of sesquiterpenes; the fatty acids of myristic acid and to a lesser extent lauric acid, with a trace of palmitic acid and a little oleic acid. In addition to otobite the authors isolated another compound iso-otobite from the unsaponifiable matter.—W. G.

*Annatto in fats and oils; Test for —.* W. Brinsmaid. J. Ind. Eng. Chem., 1921, 13, 216—217.

FIFTEEN g. of the melted and filtered fat is shaken with 15 c.c. of chloroform and 15 c.c. of 5% sodium hydroxide solution, and the mixture then heated at 50°—60° C. until the emulsion is broken up; the soap froth is transferred to a beaker, 10 c.c. of water, 2 c.c. of the sodium hydroxide solution, and a small quantity of paper pulp are added, the mixture is heated on a steam-bath for 30 mins.,

and the pulp then collected in a Gooch crucible. As soon as the liquid has passed through, the pulp is treated with a few drops of stannous chloride solution; if annatto is present a pink coloration is obtained.—W. P. S.

*Cottonseed oil; Catalytic hydrogenation of* —. J. Kahlenberg and G. J. Ritter. *J. Phys. Chem.*, 1921, 25, 89—114.

LABORATORY experiments indicate that of the common single metal catalysts, nickel is the most efficient, cobalt and iron possessing reduced activity in the order named. A 50% nickel-cobalt catalyst is more active than either metal used alone, but iron-cobalt and iron-copper possess little activity. Zinc catalysts prepared by reduction of the carbonate show some activity as also does a catalyst prepared from zinc carbonate precipitated in the presence of aluminium powder. Zinc and powdered charcoal, and zinc and pumice are not such good catalysts as zinc alone, and aluminium, zinc-aluminium prepared from zinc nitrate, and granulated zinc show no catalytic activity. On the other hand, "replaced" nickel obtained by the action of pure granulated zinc on nickel chloride solution and also nickel obtained by reduction of powdered nickel chloride with hydrogen are good catalysts, showing optimum activity of 180° C. A bismuth-charcoal catalyst is fairly active.—A. de W.

*Unsaturated fatty acids; Products of autoxidation of* —. W. Fahrion. *Chem. Umschau*, 1921, 28, 5—7, 20—21.

A SIMPLIFIED modification of Hazura's method for the detection of linolic acid in oils is given as follows: 10 g. of the fat is oxidised in the usual way with 10 g. of potassium permanganate, the alkaline solution acidified, and the separated oxidation products boiled with 1 l. of water. Any dihydroxystearic acid is filtered off from the hot liquid, the filtrate made weakly alkaline, concentrated to 100—150 c.c., and extracted with ether after acidification with hydrochloric acid. Azelaic acid, together with any unoxidised fatty acids, pass into the ethereal solution, any sativic (tetrahydroxystearic) acid floating as white flocculi in the intermediate layer. On examining cottonseed oil by this method, 10 g. of the oil gave 0.6 g. of an ether-insoluble residue, which on recrystallisation yielded white silky needles of m.p. 152° C., having an elementary composition corresponding to tetrahydroxystearic acid. Neither tallow nor mowrah fat contains linolic acid, but a fair amount is indicated as being present in lard. Goldschmidt and Weiss (*Chem. Umschau*, 1917, 158) have worked out a method for the evaluation of "fatty acids suitable for soap-making" in oxidised oils, by determining the difference between the proportion of fatty acids soluble in ether and those insoluble in petroleum spirit of high boiling-point. The oxidised acids from fish oils, however, are often to a great degree insoluble in ether. The author points out that in consequence of the partial insolubility of oxidised fatty acids in water, a further yield of these acids is obtained by extraction of the evaporation residue from the aqueous acid layer after the acidification of the soap solution. Examination of this residue insoluble in ether led to negative results for the presence of glycerin, which is thus assumed to have been volatilised during analysis or decomposed by acid. On examination of a sample of linseed oil linoxyn (sp. gr. 0.973) the following results were obtained: Hehner value, 82.9; solid oxyacids (+ glycerin?), 7.0; liquid oxyacids (by extraction of evaporated acid aqueous layer with ether), 4.6; volatile fatty acids, 4.1; moisture, 2.5; ash, 0.5. On fractionating the acids soluble in petroleum ether by the lead salt-ether method and allowing for the increase in weight due to oxidation of the oil, 6.7%

of saturated fatty acids of iodine value 12.0, were calculated on the original (unoxidised) linseed oil, a figure in substantial agreement with commonly observed values.—A. de W.

*Bird-lime; Composition of* —. Y. Nishizawa. Tokyo Kwagaku Kwai Shi (*J. Tokyo Chem. Soc.*), 1920, 41, 1043—1048. (*Cf. Divers and Kawakita, Chem. Soc. Trans.*, 1888, 268.)

FROM the saponification products of bird-lime, obtained by heating with alcoholic caustic soda for 10 days on the water bath, palmitic acid and elaidic acid were isolated.—K. K.

*Coconut oil; Determination of* — in soaps by the *Polenske method*. R. Jungkunz. *Seifensieder. Zeit.*, 1920, 47, 927—929, 949—951. *Chem. Zentr.*, 1921, 92, 11, 418—419.

A KNOWLEDGE of the Polenske value determined under proper conditions renders possible the determination of the coconut oil content of soap; difficulty is encountered, however, in distinguishing between coconut oil and palm-kernel oil. After the estimation of the fatty-acid content, sufficient soap to yield 4.75 g. of fatty acid is heated with 20 g. of glycerin and 1 c.c. of sodium hydroxide solution (1:1) over a small flame for about 10 min. until the mixture no longer froths greatly; after cooling to 80°—90° C., 90 c.c. of water, 50 c.c. of dilute sulphuric acid, and about 0.6 g. of coarse pumice stone are added, the subsequent distillation and further procedure being as usual.—D. F. T.

*Soaps; Ultramicroscopic structure of* —. W. F. Darke, J. W. McBain, and C. S. Salmon. *Proc. Roy. Soc.*, 1921, A 98, 395—409.

CURDS of sodium soap consist of a felt of hydrated fibres, enmeshing and in equilibrium with a soap sol or gel of definite concentration, the solubility rising rapidly with temperature. The individual fibres may be many centimetres long, but they are barely of microscopic diameter. Potassium soap solutions on cooling first develop fibres which are similar to those of sodium soaps, except that they are only a few hundredths of a millimetre in length and have a strong tendency to form twins. The stable condition at ordinary temperature is, however, the formation of innumerable tiny lamellar crystals of hydrated soap. The hydrogen soap, cetyl sulphonic acid, is similar to the potassium soaps, but the particles of colloidal cetyl sulphonic acid are very much more prominent.—J. F. S.

*Saponification; Time factor in* —. P. J. Fryer. *Analyst*, 1921, 46, 87—90.

As measured by the amount of alkali removed from the reacting solution, the velocity of saponification of fats and oils is in inverse ratio to the molecular weights of the fatty acids of the glycerides; in terms of the weight of fat or oil used, the velocity of saponification under the same conditions appears to be the same for all glycerides and probably for all esters. The velocity is influenced considerably by small differences of temperature, is increased by an increase in the concentration of the alkali, and also increases in direct proportion the molecular weight of the solvent used.—W. P. S.

*Petroleum from fish oils.* Kobayashi. See 11a.

*Hydrolysis of triacetin.* Yamasaki. See XX.

#### PATENTS.

*Oil, fat and the like; Recovering and utilising solvent from the air which passes away from apparatus for extracting* — by means of a solvent. J. W. Melton and C. Downs. *E.P.* 159,089, 29.11.19.

THE mixture of air and vapour leaving the con-

denser or dephlegmator of the apparatus passes into the top of a chamber containing a supply of unextracted material supported on a tray with a perforated bottom. The material absorbs the solvent from the mixture, and when it is almost saturated the supply of gases is diverted to another similar chamber. The material in the first chamber is discharged at the bottom and conveyed to the extraction plant.—L. A. C.

*Hydrogenating catalysts; Preparation of* —. C. Ellis. U.S.P. 1,369,013, 22.2.21. Appl., 5.4.16.

A MIXTURE of oil and a substantially non-catalytic reducible nickel compound is subjected to the action of a gaseous reducing agent.—A. de W.

*Copra; Recovery of oil from* —. C. O. Phillips. U.S.P. 1,369,265, 22.2.21. Appl., 17.5.19.

GROUND copra is intimately mixed with a small amount of a dilute alkaline solution and subjected to a heating and pressing operation.—A. de W.

*Soap.* C. S. Townsend. E.P. 159,083, 7.1.20.

A TOILET soap contains from 5 to 20% of the solids obtained by evaporating the waste whey derived from cheese factories.—L. A. C.

*Cooling fatty emulsions.* E.P. 134,815. See XIXA.

*Base for salves.* G.P. 329,605. See XX.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Radio-active luminous pigments; Loss of activity of* —. G. Berndt. Z. tech. Phys., 1920, 1, 102—107. Chem. Zentr., 1920, 91, IV., 717.

EXPERIMENTS conducted with a series of radio-active pigments composed of zinc sulphide containing 0.2—0.02 mg. radium per grm., showed that the loss of activity after 9 months agreed with Rutherford's formula,

$$I_t/I_0 = 1/(A.t)(1 - e^{-A.t}),$$

where  $I_0$  is the original activity and  $I_t$  the activity after time  $t$ , whilst  $A$  is a constant the value of which depends upon the experimental conditions. It was found, however, that the variations of the values for  $A$  with the radium content are more correctly represented by a hyperbola than by a straight line. Loss of activity is slower in the case of pigments prepared by diluting a highly active preparation with dry Sidot blende than in the case of pigments of equal activity prepared directly from a mixture of zinc sulphide and an aqueous radium solution. Pigments of which the activity is due to mesothorium and radiothorium depreciate to about the same extent as those containing radium. On the other hand, loss of activity from radiothorium preparations is more rapid.—W. J. W.

*Siam benzoin.* Reinitzer. See XX.

*Wormseed oil.* Langer. See XX.

#### PATENTS.

*Lake [pigment].* A. Linz, Assr. to Ultra-Chemical Corp. U.S.P. 1,369,252, 22.2.21. Appl., 9.10.20.

THE dyestuff obtained from diazotised *p*-amino-phenol and 2-naphthol-3,6-disulphonic acid is treated with a compound of a multivalent metal.

—A. de W.

*Lithopone; Process for drying* —. J. Rudolf. G.P. 329,711, 28.9.18.

LITHOPONE, direct from the precipitation vessels, without dewatering, is transferred to a revolving tube dryer coated with aluminium or zinc.

—A. R. P.

*Acid-resisting material; Preparation of an — impervious to water.* J. Frenz. G.P. 329,824, 26.2.19. Addn. to 321,029 (J., 1920, 659 A).

A SOLUTION, prepared hot and subsequently cooled, of a natural or artificial resin, bitumen, or wax, in a solvent, such as alcohol, petroleum ether, or benzol, is added to a mixture of absorbent material and a binder impregnated with paraffin to form a product suitable for application as an acid-resisting paint.—L. A. C.

*Condensation products from phenol and formaldehyde; Process for making light-coloured air- and light-proof* —. F. Pollak, Assr. to The Chemical Foundation, Inc. U.S.P. 1,369,352, 22.2.21. Appl., 12.6.14. Renewed 2.7.19.

See E.P. 20,977 of 1914; J., 1915, 1154.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Rubber; Solubility of crystalline substances in* —. G. Bruni. Giorn. Chim. Ind. Appl., 1921, 3, 51—53.

IN experiments on acceleration of vulcanisation by means of azobenzene, the author found that admixture of 10% of the latter with rubber yields a transparent, orange-red jelly, which gradually becomes opaque owing to deposition of crystalline azobenzene. Thermal analysis of the binary systems formed by rubber (freed from resin) with azobenzene, naphthalene, and *p*-toluidine shows that the rubber acts towards the crystalline constituent as an ordinary solvent, forming true saturated solutions; supercooling and supersaturation readily occur. The colloidal character of rubber plays no part in these phenomena. The marked concavity towards the concentration axis of the curves connecting temperature and composition indicates the formation of complexes between the rubber and the crystalline components of the systems. Prolonged heating of the systems produces no permanent alteration in the molecular state of the rubber, which behaves quite normally when subsequently separated from the crystalline compounds.

—T. H. P.

#### PATENTS.

*India-rubber; Compositions containing* —. W. H. Perkin, J. H. Mandelberg, and J. Mandelberg & Co. E.P. 159,014, 18.11.19.

RUBBER material is rendered non-inflammable by incorporating with it a large proportion of hydrated aluminium oxide. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 125,622; J., 1919, 409 A.)—D. F. T.

*Rubber; Treatment of raw — when freshly coagulated from the latex.* S. C. Davidson. E.P. 159,106, 19.4.20. Addn. to 151,344 (J., 1920, 757 A).

THE pin plate, fitted with a relatively small number of pins for the perforation of the rubber, is made capable of up-and-down movement, whilst the frame carrying the layer of rubber synchronously moves forward with each upward movement of the plate; in this way a fresh portion of the surface becomes perforated at each stroke until the whole has been treated.—D. F. T.

*Rubber goods; Ingredients for soft and hard* —. Farbenfabr. vorm. F. Bayer und Co. G.P. 303,224, 3.10.16.

THE oily or resinous products obtained by the condensation of halogen derivatives of alkylbenzenes, in which the halogen atom is present in the side-chain, with naphthalene and its derivatives or with



coal tar oils or their individual hydrocarbon constituents, can be used with advantage in compounded raw rubber which is difficult to manipulate on account of its "dryness" or which yields hard or brittle products on vulcanisation.—D. F. T.

*Rubber; Production of materials resembling* — H. O. Traun's Forschungslaboratorinn G.m.b.H. G.P. 329,593, 1.11.18.

BUTADIENE, homologous or analogous substances, mixtures of these, or products of their incomplete polymerisation are subjected to a pressure of 50–600 atm. at the ordinary temperature, the presence of a solvent or of a polymerisation catalyst being optional. With butadiene itself under a nitrogen pressure of 300 atm. the formation of butadiene-rubber is complete in about 4 weeks, whilst with a pressure of 600 atm. the period is only 5–6 days.—D. F. T.

*Vulcanised rubber; Process for devulcanising* — C. F. Willard. E.P. 159,987, 9.12.19.

SEE U.S.P. 1,322,152 of 1919; J., 1920, 74 A.

*Treating leather.* U.S.P. 1,369,240. See XV.

## XV.—LEATHER; BONE; HORN; GLUE.

*Gallotannic acid; Extraction of* — E. Knappe. Chem.-Zeit., 1921, 45, 239–241.

GALLS are swollen with water, ground, and extracted with ether or alcohol for 20–30 mins. in a cylindrical shaker fitted with an agitator. The extract is filtered off and three more extractions made. A clearer, brighter solution is obtained in this way than from dry powdered galls, and less solvent is required. Saturating the solvent with water and using this for the extraction of powdered dry galls is not so successful. The ether may be replaced by trichloroethylene or dichloroethylene with equally good results.—D. W.

[*Tannin analysis.*] *Comparison of the international filter-tube method and the official hide-powder method.* H. W. Wiley and W. H. Krug. J. Amer. Leather Chem. Assoc., 1920, 15, 51–53.

SAMPLES of valonia, mimosa bark, oakwood, chestnut, mimosa, and quebracho extracts were analysed by the filter-tube method in England and by the official method in America. The results were compared and show good agreement for moisture and "reds" except with the quebracho extract, where the filter-tube method gives higher results. In every case except one, the filter-tube method gives lower values for non-tannins than the official method and correspondingly higher values for tannin.—D. W.

*Sole leather. Sole and upper leathers for army boots.* C. Schiaparelli. J. Soc. Leather Trades Chem., 1921, 5, 44–56.

THE normal hygroscopicity of sole leathers is about 4%, but the presence of hygroscopic substances, e.g., glucose, may increase it to 7–8%. The rigidity of the leather can be determined by cutting samples of standard length and thickness, clamping one end, and noting the depression caused by weighting the other end with a standard weight. The amount of water-soluble matter as determined by the Koch extractor is not comparable with the results of actual wear. Tests should be made by immersing whole soles in water for 72 hrs., and results show that there seem to be three kinds of water-soluble matter in unadulterated sole leathers, viz., an easily soluble portion, a less easily soluble part, and 3% of difficultly soluble matter extracted only in the Koch extractor. The permeability is determined by immersing weighed portions of leather in water for 24 hrs., then re-weighing and

adding to the increase the weight of the soluble matter extracted by the water during immersion. The purer the tannage and the less the natural soluble matter the leather contains, the more easily it is penetrated by water. The permeability is an inverse function of the percentage of water-soluble matter. There are two kinds of sole leather, viz., leather of the English type or valonia-tanned leather, with high content of water-soluble matter for damp, cold climates, and leather with a low content of water-soluble matter for hot dry climates.—D. W.

## PATENTS.

*Leather; Method of treating* — S. O. Hahn, Assr. to The Chrome Leather and Rubber Tyre Co. U.S.P. 1,369,240, 22.2.21. Appl., 16.2.20.

A PORTION of the sulphur is removed from ordinary commercial rubber cement, the product is forced into the leather beyond the surface, and the impregnated leather is subsequently united to a rubber layer by vulcanisation.—D. W.

*Drying solutions.* G.P. 331,142. See I.

## XVI.—SOILS; FERTILISERS.

*Soils; Degree of temperature to which — can be cooled without freezing.* G. Bouyoucos. J. Agric. Res., 1920, 20, 267–269.

A COLUMN of wet soil was placed in a freezing point tube, the bulb of a Beckmann thermometer inserted into the soil, and the tube placed in mixtures at different low temperatures until a temperature was found at which the soil froze spontaneously. All mineral soils could be cooled to  $-4.2^{\circ}\text{C}$ . and kept at that temperature indefinitely without freezing. Agitation of the soil however caused it to freeze at once. The amount of water in the soil made no difference. Peat and "muck" could be supercooled to  $-5^{\circ}\text{C}$ . Some other materials, such as silica, carbon black, and gelatin, saturated with water, were tested also, and these could be cooled to  $-6^{\circ}\text{C}$ . without freezing. The experiments show that in winter when the temperature of the soil falls only a few degrees below zero, the soil does not really freeze.—J. H. J.

*Soil; Effect of various crops upon the water extract of a typical silty clay loam* — G. R. Stewart and J. C. Martin. J. Agric. Res., 1921, 20, 663–667. Cf. J., 1918, 252 A.)

THE effect of crops of maize, horse beans, potatoes, turnips, and barley upon the water extract of a loam soil was studied throughout the growing season. All the crops reduced the concentration of the water extract of the soil during the height of the growing season, as judged by the change in electrical conductivity of the extract. In another series of experiments, the concentration of the soil solution was determined in the neighbourhood of the plant roots and in a part of the same ground without plants. The observations were made by the method of freezing point determinations. No movement of solution towards the plants was observed, and no change in concentration occurred in the unplanted soil until the plant roots reached that area.—J. H. J.

*Potassium; Concentration of — in orthoclase solutions not a measure of its availability to wheat seedlings.* J. F. Breazeale and L. J. Briggs. J. Agric. Res., 1921, 20, 615–621.

FINELY ground orthoclase containing 12.5%  $\text{K}_2\text{O}$  was shaken with distilled water, and germinated wheat seeds were floated on the surface of the mixture. The liquid contained 2–9 pts. of dissolved potash per million pts. The amount of potash in a



hundred seedlings after 2–3 weeks' growth was determined and was compared with controls in solutions free from potash. The results showed that the seedlings did not absorb any potassium from the solutions, although from potassium chloride solutions of the same concentration absorption took place readily. Addition of lime, gypsum, and carbon dioxide to the solutions did not render the potassium available, nor did boiling the solution. On the other hand, from a solution which had been filtered and oxidised by evaporation with acid, large amounts of potash were absorbed by the plant, and similar results were obtained with solutions which had been filtered through a Pasteur-Chamberland filter to remove colloids and then oxidised. The potassium in orthoclase thus appears to be combined in a complex form which is soluble in water, but is not available for plant use. The inference is drawn that the concentration of a plant food in a soil solution is not necessarily the measure of its availability to the plant.—J. H. J.

*Soluble phosphate in superphosphate; Conditions affecting the reversion of* —. B. Neumann and K. Kleylein. *Z. angew. Chem.*, 1921, 34, 77–80, 84–86.

In an examination of the influence of various reversion agents, preliminary experiments were carried out with pure monocalcium phosphate and phosphoric acid. Excess of calcium sulphate, or the presence of unconverted tricalcium phosphate, even at normal temperature, is instrumental in promoting decomposition of the soluble phosphate. If iron and aluminium exist as sulphates, their reverting action is not appreciable. Thus, after addition of these two sulphates, even up to 16%, to monocalcium phosphate, the total soluble phosphate found was 89.28% and 99.68%, respectively, and the loss with phosphoric acid was still less. On the other hand, considerable reversion occurs if the oxides of these metals are present in sufficient excess to escape conversion into sulphate. An addition to monocalcium phosphate of 4% of iron oxide reduced the soluble phosphate to 81.82%, and in the case of aluminium oxide, to 78.56%; with 16% of these oxides, the amounts of soluble phosphate were only 47.75% and 51.15%, respectively. The action is less marked with phosphoric acid, and its presence in superphosphate therefore to some extent mitigates loss of soluble phosphate. In commercial superphosphates, the effect of adding excess of sulphuric acid, in order to convert ferric oxide into sulphate, only partially achieves its object, an increased yield of about 4.5% of soluble phosphate being obtained with an increase of 12% of acid, and the presence of free acid in the resultant superphosphate renders the use of a large excess of acid undesirable. If the raw material contains more than 2% of ferric oxide, it is preferable to mix it with phosphates of smaller iron content. Aluminium compounds in commercial phosphates have no influence on the yield of soluble phosphate; the aluminium phosphate which is formed appears to be retained in a soluble condition by the free acid which is present.—W. J. W.

#### PATENTS.

*Cheese manufacture; Treatment of whey produced in —, and production therefrom of nitrogenous matter suitable for use as a fertiliser.* J. Taveras, J. W. Roche, and G. Martin. E.P. 158,816, 22.7.20.

The waste whey is heated to about 70° C. and agitated, the albumin present is then precipitated by addition of bleaching powder (about 0.002%), and the precipitate is collected and mixed with quicklime.—J. H. L.

*Boiling and drying organic substances; Apparatus for* —. K. Niessen. E.P. 137,841 and 137,844, 13.1.20. Conv., 20.7.16 and 12.5.17. Addns. to 137,828.

SEE G.P. 319,335–6; J., 1920, 498 A.

*Urea; Compound of* —. [Fertiliser.] C. Bosch, Assr. to The Chemical Foundation, Inc. U.S.P. 1,369,383, 22.2.21. Appl., 27.4.16.

SEE G.P. 295,548 of 1915; J., 1917, 560.

#### XVII.—SUGARS; STARCHES; GUMS.

*Sugar; Suggestions on the production of a better raw* —. S. S. Peck. *Int. Sugar J.*, 1921, 23, 158–161.

By the use of screens of relatively large mesh for straining the raw juice, a greater or less amount of very fine particles of cane fibre (*bagacillo* or “cush-cush”) is permitted to pass through, so that during the subsequent operations of liming and heating the gum content becomes increased, owing to the solution of the pentosans and lignin, of which the fibre is largely composed. Although the gum content of the juice is normally small (viz., 2–5% of the sucrose present), its influence is important, particularly in retarding the boiling, crystallising, and centrifuging of the low-grade products. The use of a 100-mesh screen for the separation of the greatest amount possible of *bagacillo* from the raw juice previous to clarification is recommended.

—J. P. O.

*Sugars; Deterioration of — in storage.* N. Kopcloff, H. Z. E. Perkins, and C. J. Welcome. *J. Agric. Res.*, 1921, 20, 637–653. (Cf. J., 1920, 380 A, 523 A, 553 A.)

A LARGE number of bags of raw sugar was examined chemically and bacteriologically at intervals of 4 weeks under ordinary conditions of storage. The period of observation was 8 weeks and there was found to be a loss in polarisation in most samples with a gain in reducing sugar and an increase in moisture. The number of bacteria and moulds increased in most cases, the numbers being greatest in those samples which suffered most deterioration. A large initial infection led to the greatest deterioration. When the number of organisms present was below 200 per g. and the moisture ratio was low, then the deterioration was either greatly reduced or prevented altogether.—J. H. J.

*Decolorising carbons [for sugar refining]; Comparative value of* —. F. E. Thomas. *Int. Sugar J.*, 1921, 23, 162–165.

A STANDARD method of testing decolorising carbons for use in sugar manufacture and refining is recommended, and it is pointed out that the colour remaining in the treated liquid is of greater importance than that removed, and that different carbons adsorb the several colouring matters in varying degree. In carrying out the test, raw sugar liquor at 50° Brix, having a slightly acid reaction, is mixed in the cold with the sample of carbon (about 5% of the sugar present), raised to boiling point, and filtered, a standard carbon (preferably “Norit,” owing to its great uniformity of efficiency) being treated under exactly the same conditions. An error is produced when observing the degree of colour in the Lovibond tintometer if the untreated and treated solutions are read in cells of different size, e.g.,  $\frac{1}{2}$  and  $\frac{1}{4}$  respectively, as is frequently done.—J. P. O.

*Sucrose; An inherent error in certain modifications of the Clerget method of determining — by double polarisation.* C. A. Browne. *Int. Sugar J.*, 1921, 23, 166–167.

IN the so-called neutral method of double polarisa-

tion employed by Jackson and Gillis (J., 1920, 634 A), the influence of sodium chloride, ammonium chloride, etc., upon the rotation of the invert sugar, and of amino-acids and other optically-active non-sugars, will depend, not only upon the amount of salt present in 100 c.c. of solution, but also upon the amount of water and sugar in the solution. Thus, solutions containing 52, 26, and 13 g. of sucrose in 100 c.c., polarised 99°00', 99°35', and 99°50' (calculated to 26 g.), though the amount of sodium chloride, viz., 2.315 g., present in each was the same. Similar results were obtained in the case of ammonium chloride, thus demonstrating that as the amount of sucrose in 100 c.c. diminishes, and the corresponding amount of water increases, the action of the dissolved salt upon the rotation of the sugar becomes correspondingly less, and the values approach 100. Therefore the fixed values of sucrose + 2.315 g. NaCl = 99°38', and + 3.392 g. NH<sub>4</sub>Cl = 99°43', employed by Jackson and Gillis as the positive constituents in their evaluation of the Clerget divisor, are true only for a concentration of 26 g. of sucrose in 100 c.c.—J. P. O.

**Maize sugar.** P. Vieillard. Bull. Agr. Inst. Sci. de Saigon, 1920, 2, 106. J. Inst. Brew., 1921, 27, 126.

THE stem of the maize plant normally contains no sucrose and very little dextrose, but if the ears are removed when the grain begins to be milky, sugars accumulate in the stem. In a series of experiments the stems were investigated at various periods (7—34 days) after removal of the ears. The stems freed from leaves yielded 36—48% of juice, as compared with 80% in the case of the sugar cane, but the "bagasse" contained much sugar extractable by suitable means. The total extractable sugar in the stems amounted to 7—10% of sucrose and 1—3% of dextrose. Owing to the large proportion of dextrose and non-sugars not more than one-third of the sucrose present can be obtained crystalline, but for the production of alcohol this is immaterial. It is calculated that 130 galls. of alcohol (absolute) could be produced per acre of maize crop, and the period between the sowing of the maize and the gathering of the stems would be about 100 days.

—J. H. L.

**Maltose or lactose; Determination of — in the presence of other reducing sugars.** Legrand. Comptes rend., 1921, 172, 602—604.

BARFOED's solution (5 c.c. of 38% acetic acid added to 200 c.c. of a solution of 1 pt. of normal copper acetate in 15 pts. of water) is reduced by hexoses but not by disaccharides. Hexose sugars are estimated by boiling 5 c.c. of the sugar solution, containing not more than 0.1 g. of sugar, with 15 c.c. of Barfoed's solution for 3 mins. in a conical flask. The cuprous oxide is collected and estimated volumetrically as in Bertrand's method (J., 1907, 60). Total reducing sugars are then estimated by one of the usual methods with Fehling's solution.

—W. G.

**Rhamnose; Preparation of —.** C. F. Walton, jun. J. Amer. Chem. Soc., 1921, 43, 127—131.

THE commercial product "Lemon Flavin" is rich in quercitrin and forms an excellent starting material for the preparation of rhamnose. The flavin is boiled for 30 mins. with 10 pts. of 0.5% sulphuric acid. The residue consisting of quercetin is filtered off and washed free from sugar, and the combined filtrates are neutralised with barium carbonate, decolorised, and concentrated under diminished pressure to a density corresponding to about 40% of solids. The liquid is then treated with 3 vols. of warm absolute alcohol, filtered, and the filtrate concentrated to a density corresponding to 70—80% of solids. On cooling the rhamnose crystal-

lises out and may be purified by recrystallisation from 80% alcohol. A 20—25% yield is obtained.

—W. G.

**Colloids; Plant —. IX. Various starches.** M. Samec and H. Haerdtl. Koll. Chem. Beihefte, 1920, 12, 281—300.

ALL varieties of starch can be separated by electro-dialysis into a highly viscous and electro-conducting fraction (Meyer's  $\beta$ -amylose, Maquenne's amylopectin) and a non-viscous and non-conducting fraction (Maquenne's amylose). The amounts of these constituents vary from starch to starch, and dependent on the ratio of these amounts the water content of the starch granules varies as well as the viscosity and resistance to diastase of the solutions. All varieties of starch contain phosphorus; the following amounts per 100 g. of starch were found: potato, 0.112 g.; meadow saffron (*Colektion autumnale*), 0.016 g.; maranta, 0.031 g.; tapioca, 0.012 g.; turmeric, 0.162 g.; horse-chestnut, 0.016 g.; wheat, 0.105 g.; maize, 0.034 g.; and rice, 0.039 g. The mean molecular weight varies with the different varieties thus: potato, 123,000; meadow saffron, 231,000; maranta, 260,000; tapioca, 208,000; turmeric, 158,000; horse-chestnut, 230,000; wheat, 170,000; *Oryza glutinosa*, 173,000; maize, 77,500; and rice, 110,000. (Cf. J.C.S. April.)—J. F. S.

**Starch; Polarimetric determination of —.** H. Lüthrig. Pharm. Zentralh., 1921, 62, 141—144.

THE effect of slight modifications of procedure in Ewers' method (J., 1908, 238) were investigated; the trustworthiness of the method is not affected by slight alteration in the strength of the acid used or in the treatment before and after the addition of the acid, but the time of heating prescribed (15 mins.) should not be exceeded. A method described by Mannich and Lenz (Z. Unters. Nahr. Genussm., 1920, 1), in which the starch is dissolved by heating with calcium chloride solution containing a small amount of acetic acid, and then polarised, also yields trustworthy results.—W. P. S.

**Potash in molasses.** Sherrill. See XXIII.

#### PATENTS.

**Sugar-washing machine; Continuous —.** E. C. Carrick, Assr. to N. A. Lockwood. U.S.P. 1,367,946, 8.2.21. Appl., 19.1.20.

AN endless movable filtering surface forms the peripheral wall of a series of chambers, which travel around a central axis. Means are provided for producing a vacuum within the chamber in the uppermost position with its filtering surface, on which the sugar is placed, at the top, so that the filtrate is drawn into the chamber, and for producing a pressure within the chamber in the lowest position with the filtering surface at the bottom, whereby the filtrate is expelled through the filtering surface. Means are also provided for removing the sugar from the filtering surface after the chambers have passed the vacuum zone and before they have entered the pressure zone.—J. H. L.

**Karaya gum; Process of producing dry water-soluble products from —.** H. V. Dunham. E.P. 160,045, 20.1.20.

SEE U.S.P. 1,334,356—8 of 1920; J., 1920, 380 A.

#### XVIII.—FERMENTATION INDUSTRIES.

**Malts; Iodometric determination of the diastatic power of —.** J. L. Baker and H. F. E. Hulton. Analyst, 1921, 46, 90—93.

THE method described depends on the oxidation of maltose to maltohionic acid by iodine in alkaline

solution. Varying quantities (1, 2, or 3 c.c., etc.), of malt extract are allowed to act for 1 hr. at 21° C. on 100 c.c. of 2% soluble starch solution; action is then stopped by the addition of 10 c.c. of *N*/10 sodium hydroxide solution, and the mixture is diluted to 200 c.c. Fifty c.c. of this mixture is treated with 20 c.c. of *N*/10 iodine solution and 30 c.c. of *N*/10 sodium hydroxide solution; after 10 mins. 4 c.c. of *N*/1 sulphuric acid is added, and the excess of iodine is titrated with *N*/20 thiosulphate solution. Under these conditions the diastatic power =  $16 \cdot 7Y/X$ , where *Y* = c.c. of *N*/10 iodine solution used for the oxidation and *X* = c.c. of 5% malt extract taken for the conversion.—W. P. S.

*Yeast; Nutritional requirements of* —. I. *Role of vitamins in the growth of yeast*. E. I. Fulmer, V. E. Nelson, and F. F. Sherwood. J. Amer. Chem. Soc., 1921, 43, 186—191.

CULTURE experiments with extracts of alfalfa and wheat embryo show that water-soluble-B vitamin is not essential for the growth of yeast.—W. G.

*Yeast; Nutritional requirements of* —. II. *Effect of the composition of the medium on the growth of yeast*. E. I. Fulmer, V. E. Nelson, and F. F. Sherwood. J. Amer. Chem. Soc., 1921, 43, 191—199.

YEAST was grown satisfactorily for three months on a synthetic medium of inorganic salts and sucrose, devoid of calcium and magnesium. The optimum concentration of several ammonium salts examined was the same and corresponded to the concentration of ammonium chloride in which there was the minimum swelling of protein as measured by wheat gluten. The presence of certain colloids, such as dextrin, favoured the growth of the yeast.—W. G.

*Fermentation; Water-soluble vitamin and substances accelerating* —. I. *Method for the determination and preparation of a substance from yeast and rice polishings which accelerates fermentation*. S. Fränkel and E. Schwarz. Biochem. Zeits., 1920, 112, 203—235.

UTILISING the fact that extracts from yeast and rice polishings accelerate fermentation, the authors have worked out a method by means of which the accessory factor could be estimated. A fraction from yeast was prepared, having an activity 22 times greater than that of the alcoholic extract of the yeast, by successive precipitations with basic lead acetate, mercuric chloride, and phosphotungstic acid, this method giving better results than direct precipitation of the extract with phosphotungstic acid. (Cf. J.C.S., 1921, ii., 228.)

—S. S. Z.

*Enzymes; Chemistry of* —. T. Bokorny. Allg. Brauer- u. Hopfenzeit., 1920, 705—706, 713—714. Chem. Zentr., 1921, 92, I., 372. (Cf. J., 1920, 202A.)

NITROGEN is evolved on treatment of enzymes with nitrous acid, indicating the presence of amino-groups. The activity of emulsin is not destroyed by a 1% solution of nitrous acid. Whilst sulphurous acid is much more toxic than sulphuric acid to living organisms, possibly owing to the presence of free aldehyde groups in living protoplasm, there is little difference in the toxicity of the two acids towards enzymes; 1% solutions of both acids destroy the activity of emulsin, but 0.1% solutions have only a slightly injurious effect. By treatment of 2 g. of pressed beer yeast with 2 c.c. of 0.5% sulphuric acid, or 0.015 g. of formaldehyde, the yeast was killed without any considerable loss of zymase activity, whilst 3 c.c. of the acid or 0.25 g. of formaldehyde destroyed life and fermenting power: 0.005 g. of mercuric chloride killed 10 g. of yeast without destroying its fermenting power. Sucrose

is fermented somewhat more rapidly than dextrose, and maltose about as rapidly as the latter. It is suggested that the dextrose liberated by hydrolysis of these disaccharides is more rapidly fermentable in the nascent state than in its usual form.

—J. H. L.

*Enzymes; Nature of oxidising and reducing* —. F. Battelli and L. Stern. Comptes rend. Soc. Biol., 1920, 83, 1544—1545. Chem. Zentr., 1921, 92, I., 332—333.

NUMEROUS experiments on the tissues of higher animals revealed a close parallelism between the oxidising power of the tissues in presence of molecular oxygen and their power of decolorising thionin by converting it into the leuco-base. The results confirm Wieland's hypothesis of the identity of oxidising and reducing enzymes. The action of oxidising enzymes may be regarded also as similar to that of hydrolytic enzymes, but in the latter case the two ions of water combine with the same molecule, whilst in the former the hydrogen ion combines with one molecule and the hydroxyl ion with another.—J. H. L.

*Enzyme action; Relation of pressure and temperature to* —. I. *Influence of pressure on the velocity of peptic, tryptic, and diastatic hydrolysis*. S. Fränkel and G. Meldolesi. Biochem. Zeits., 1921, 115, 85—95.

DURING the first two hours of peptic digestion there is more protein hydrolysed when the pressure is high than when the digestion is carried out under ordinary pressure. The acceleration of digestion is less at pressures above than at pressures below 5 atm. The acceleration in the velocity of hydrolysis during the first 2 hrs. effected by high pressure diminishes eventually. In the cases of trypsin the increase in the rate of digestion is more regular. Diastatic hydrolysis is accelerated by increased pressure during the first 2 hrs., after which time it becomes slower and then remains constant.

—S. S. Z.

*Fermentation of worts containing added sugars*. J. O'Sullivan. J. Inst. Brew., 1921, 27, 93—100.

FERMENTATIONS carried out with a large number of single-cell cultures prepared from three brewery yeasts indicated no appreciable differences in fermenting power. In experiments with a malt wort of sp. gr. 1.061, treated with sucrose, invert-sugar, dextrose, or maltose equivalent to 15% of the extract present, and fermented with 0.2 g. of pressed yeast for 10 days at the ordinary temperature, the whole of the added sugar, together with the fermentable matter of the wort, disappeared in all cases. In fermentations of solutions of the same concentration, but containing different proportions of fermentable sugars and the same quantity of nutrient matter, practically the whole of the fermentable sugars was fermented when the alcohol produced did not exceed 5%, but beyond this alcohol-content some of the sugar remained unfermented.—J. H. L.

*Acetic and lactic acids; Fermentation process for the production of* — from corn [maize] cobs. E. B. Fred and W. H. Peterson. J. Ind. Eng. Chem., 1921, 13, 211—213.

WHEN corn (maize) cobs are hydrolysed by heating with 0.5—2% sulphuric acid under a pressure of 15 lb. per sq. in., they yield 30—40% of their weight of xylose. The crude xylose syrup is fermented readily by *Lactobacillus pentaceticus* with the formation of acetic and lactic acids; results of small scale experiments indicate that each ton of cobs would yield at least 300 lb. of acetic acid and 320 lb. of lactic acid.—W. P. S.

*Alcohol; Rapid volumetric method for determining* — A. Lachman. J. Ind. Eng. Chem., 1921, 13, 230.

A DEFINITE weight (25 g.) of aniline is added to 50 c.c. of the alcohol-water mixture in which the alcohol is to be determined; if the aniline does not dissolve completely, some fixed volume, e.g., 25 c.c., of strong alcohol of known strength is added. Water is then run in from a burette until a permanent turbidity is produced, and the temperature of the mixture is noted; if it is kept between 14° and 17° C., a correction is unnecessary. From the known volumes of the sample, of the added alcohol, and the added water, the percentage of alcohol in the sample can be calculated; the total volume of the solvent (alcohol plus water) is a nearly strictly linear function of the volume of the contained alcohol. Tables or graphs may be prepared (from actual determinations with known quantities of alcohol), and reference to these will give the percentage of alcohol directly from the volume of total solvent.—W. P. S.

*Methyl alcohol; Determination of* — in sulphite spirit. R. Sieber. Papier-Fabr., 1921, 19, 189—192.

SULPHITE spirit is employed in Sweden for the production of potable spirits. The raw sulphite spirit contains, on the average, about 2.5% of methyl alcohol, and the brandy produced therefrom less than 0.5%. A slight modification of Denigès' method (J., 1910, 585) is recommended for the determination of methyl alcohol in the raw spirit. 1 c.c. of the ten-fold diluted sample (or more if the sample contains less than 1.5%) is mixed with 2.8 c.c. of water and 0.2 c.c. of sulphuric acid, and the cold mixture is treated with 1 c.c. of 5% permanganate solution and after 2 mins. decolorised and treated as described by Denigès. Comparative tests are made with 0.5 c.c. of a 1%, and 1 c.c. of a 0.1% solution of methyl alcohol in water or pure ethyl alcohol; if solutions in ethyl alcohol are employed 1 c.c. of permanganate solution should be used for each, but for aqueous solutions 0.5 c.c. is sufficient, water being added in all cases to bring the total volume to 5 c.c. The colorimetric comparison is made after the liquids have stood for 1 hr., or longer if possible, and Von Fellenberg's tables (J., 1915, 574) will be found useful.—J. H. L.

*Diastatic preparations.* Tagliani. See VI.

*Maize sugar.* Vieillard. See XVII.

*Oxynitrilase.* Krieble and Wieland. See XX.

#### PATENTS.

*Glycerin; Process and apparatus for the continuous distillation of* — from the residue obtained in the distillation of fermented liquids. E. Barbet et Fils et Cie. E.P. 129,649, 9.7.19. Conv., 11.7.18.

AFTER concentration to a sp. gr. of at least 1.25 in a multiple-effect apparatus, the fermentation residue is almost completely dehydrated in a suitable evaporator, and the hot syrup is then introduced into a column still, maintained under as complete a vacuum as possible, in which it is systematically exhausted by means of superheated steam, the glycerin distillate being carried over into a condenser, discharged therefrom under gravity, and subsequently concentrated. The column employed has plates with heating worms, and is provided with non-conducting or steam-jacketed walls; the steam bubbles through the liquid in the lower part of the column, and each plate is worked by superheated steam. The highly concentrated residue is dis-

charged from the column under gravity, through a steam-jacketed pipe to prevent premature solidification.—J. H. L.

*[Glycerol]; Process for manufacturing of propantriol* — from sugar. W. Connstein and K. Lüdecke. U.S.P. 1,368,023, 8.2.21. Appl., 13.8.19.

SOLUTIONS containing sugars are fermented by yeast, after addition of soluble salts of non-alkaline reaction in amounts more than sufficient for the nutrition of the yeast.—J. H. L.

*Drying apparatus.* G.P. 330,129. See I.

#### XIXA.—FOODS.

*Wheat; Changes taking place in the tempering of* — E. L. Tague. J. Agric. Res., 1920, 20, 271—275.

BEFORE grinding wheat, it is the practice to add water to bring the total moisture present up to 15.5% and to allow to stand for 12—48 hrs., with the result that the bran is toughened and more readily separated and the flour obtained in higher yield and of better colour. The principal factors involved are amount of water added, time, and temperature. Three varieties of wheat were tempered with water in a closed bottle in a thermostat for a definite time, and were then ground to flour. Water extracts of the flours were prepared, and determinations were made of the hydrogen ion concentration, the total acidity, the soluble phosphorus, and the nitrogen titratable by the Sørensen formaldehyde method. Tempering at 5° C. caused little change in the flour compared with an untempered flour. At 20° C. chemical changes took place, each of the above determinations giving higher results than with the untempered flour; time also appeared to increase the action and the yield of flour was larger. At 40° C. the changes were more pronounced, but the milling properties of the wheat had deteriorated, especially after lengthening the time. Increasing the water to 18% produced a sticky flour. The general conclusion is drawn that the improved milling quality of tempered wheat is due chiefly to physical changes.—J. H. J.

*Milk; Cryoscopy of* — J. Hortvet. J. Ind. Eng. Chem., 1921, 13, 198—203.

A DETAILED description is given of the procedure to be adopted and of apparatus recommended for use in determining the freezing point of milk. In the case of milk from individual cows, the freezing pt. varied from -0.534° to -0.562° C., whilst for mixed milk from a herd of cows it lay between -0.545° and -0.562° C.—W. P. S.

*Colloids; Fixation of mineral salts by organic* —, and the condition of the salts in some vegetables. A. Scala. Ann. d'Ag., 1920, 30, 251-273. Chem. Zentr., 1921, 92, 1, 333-334.

WHEN certain neutral salts are brought into contact with colloids such as gelatin, agar, or muscle-flesh, acids are liberated to an extent dependent on the nature and the degree of dissociation of the salts and on the nature of the colloids. This effect is attributed to the ready hydrolysis of the anion of the adsorbed salt. On continued washing neutral and then basic wash-waters are obtained provided that the metal of the salt forms a soluble hydroxide (alkalis and alkaline earths); but in the case of heavy metals and aluminium the base remains fixed to the colloid. Beetroots, potatoes, etc., contain salts combined with colloids in this way, and their behaviour on continued washing is similar to that of the gelatin-salt complexes. These complexes are very stable at 0° C., but at 50°—70° C. they are

irreversibly modified and readily yield up their acids on washing. It is suggested that vitamins and accessory foodstuffs may owe their activity to inorganic salts combined with organic substances.

—J. H. L.

*Rice; Products of putrefaction of polished* — K. Yoshimura and K. Chenfon. Nippon Kwagaku Kwai Shi (J. Chem. Soc. Japan), 1921, 42, 22—37.

THE acids produced during the putrefaction of polished rice (2 kg. of rice with 4 l. water left to stand for 10 days at 30°—33° C.), were principally lactic acid (0.6%) and possibly acetic and butyric acids (0.67% as acetic acid). The following substances were isolated from 2 kg. of rice left to stand with 2 l. water for 19 and 33 days respectively: Iminazole-ethylamine (chloroplatinate, 0.8 and 0.2 g.), putrescine (hydrochloride, 0.7 and 2.0 g.), amylamine (trace and none), and ammonia (5.5 and 5.2 g.).—K. K.

*Methyl anthranilate; Detection of* — in fruit juices. F. B. Power. J. Amer. Chem. Soc., 1921, 43, 377-381.

500 c.c. of the fruit juice is steam distilled, 200 c.c. of distillate being collected and extracted with 3 successive 10 c.c. portions of chloroform. The extract is filtered through a dry filter and the chloroform evaporated in a water bath in a current of air. The residue is at once dissolved in 2 c.c. of 10% sulphuric acid, cooled, and diazotised, the diazotised liquid being poured into 2 c.c. of alkaline  $\beta$ -naphthol solution. A yellowish-red precipitate is indicative of methyl anthranilate in the original juice, the test being sensitive to 0.0001 g. of the ester. Alternatively the diazotised liquid may be tested by the addition of dimethylaniline, but this process is not so sensitive.—W. G.

*Annatto in fats.* Brinsmaid. See XII.

*Lactose.* Legrand. See XVII.

*Enzyme action.* Fränkel and Meldolesi. See XVIII.

#### PATENTS.

*Fatty emulsions [margarine]; Method for cooling or for allowing to crystallise concentrated* — K. Erslev. E.P. 134,815, 25.9.19. Conv., 7.11.18.

A CLOSED vessel is connected at the top with an exhausting air pump and is provided with a central vertical shaft entering at the top of the vessel and terminating about midway of the height in a horizontal disc. A high vacuum is established in the vessel, and the emulsion is fed on to the upper face of the rotating disc through an inlet pipe, whereby it is sprayed into the exhausted space and thereby cooled. The cooled emulsion is removed from the bottom of the vessel by a worm conveyor. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 3555 of 1890.)—A. DE W.

*Food products; Apparatus for dehydrating* — G. A. Sykes, Assec. of F. L. Nichols. E.P. 146,917, 6.7.20. Conv., 4.2.19.

A METAL casing, open at the bottom, is provided with sloping baffle plates arranged so as to form a central flue closed at the top and tapering upwards, and two side flues closed at the bottom and tapering downwards. Trays with wire gauze bottoms for containing the material slide into slots attached to the upper edge of one baffle plate and the lower edge of the plate above. Hot air, e.g., from a stove, passes up the central flue, through the trays, and out through the side flues.—L. A. C.

*Food; Manufacture of articles of* — [with the aid of sour whey solids]. C. S. Townsend. E.P. 158,684, 10.11, 6.12, and 13.12.19.

THE dried solids of sour whey from the manufacture of cheese, are employed as an ingredient of bread, pastry, confectionery, baking powders, and the like.—J. H. L.

*Flour; Treatment of* — H. Greville. E.P. 158,917, 27.6.19.

Sodium or calcium per-metaphosphate and/or pyrophosphate, or the corresponding acids in the dry state, prepared by evaporating *in vacuo* a mixture of hydrogen peroxide and a metaphosphate or pyrophosphate, or by electrolysis of cooled solutions of a metaphosphate or pyrophosphate, are added to flour. (Cf. E.P. 124,298; J., 1919, 385 A.)

—L. A. C.

*Flour and similar meals; Method for improving* — Naaml. Vennoots. Industriële Maatschappij voorh. Noury & van der Lande. G.P. 330,694, 15.8.17. Addn. to 312,528 (J., 1919, 842 A).

By submitting flour repeatedly to rapid cooling and heating, the weight and volume of the bread obtainable from it are increased.—D. F. T.

*Cheese and process for sterilising same.* G. H. Garstin, Assr. to Phenix Cheese Co. U.S.P. 1,368,624, 15.2.21. Appl., 28.10.20.

COMMUNUTED cheese is mixed with about 5% of sodium phosphate, and sterilised. Sterilised cheese containing tertiary sodium phosphate is claimed.—J. H. L.

*Eggs; Preservation of* — E. Utescher. G.P. 323,423, 9.12.14.

THE eggs are preserved in water containing, in suspension, magnesium hydroxide, with or without the addition of calcium hydroxide.—L. A. C.

*Milk; Process and apparatus for the drying of* — in a porous form. L. Jäger. G.P. 329,215, 25.11.19.

THE milk is boiled and the froth produced is conducted into a drying apparatus.—J. H. L.

*Lupins and other leguminous seeds; Removal of bitter substances from* — by means of alcoholic solvents. C. F. Hildebrandt and B. Rewald. G.P. 329,216, 29.1.18.

THE material is extracted in the cold with aqueous methyl alcohol, or mixtures of methyl alcohol with fat solvents. Fats and bitter substances may thus be extracted from the whole seeds, after treatment with water or steam.—J. H. L.

*Foodstuffs; Extraction of* — with water or aqueous solutions. L. Engelhardt. G.P. 329,505, 17.12.18.

THE bitter constituents of lupin seeds, acorns, soya beans, or the like, are removed by alternate extraction with water or an aqueous solution and electrolytic treatment of the not too finely divided material.—L. A. C.

*Straw jodder or the like; Treatment of gases from apparatus for drying light, glutinous, absorbent material, such as* — L. Roselius. G.P. 330,186, 3.1.17.

THE hot gases from the dryer are diverted sideways in the exit shaft and sprayed with a jet of water at right angles to their path. The impact of the water and absorption of the same by the fine material in suspension cause the material to fall into a receiver below.—L. A. C.

See also pages (A) 247, *Drying apparatus* (G.P. 330,129). 248, *Separating aqueous vapours from liquids* (E.P. 159,054). 269, *Soap* (E.P. 159,083). 271, *Fertiliser from whey* (E.P. 158,816). 279, *Caffeine from tea plant waste* (E.P. 159,097).

## XIXB.—WATER PURIFICATION; SANITATION.

*Resorcinol; Preparation of some alkyl derivatives of — and the relation of their structure to anti-septic properties.* T. B. Johnson and F. W. Lane. J. Amer. Chem. Soc., 1921, 43, 348—360.

THE length or weight of the alkyl group introduced into the nucleus of resorcinol in a position ortho to one hydroxyl group and para to the other has a very marked influence in increasing the antiseptic value of resorcinol. The ethyl, *n*-propyl, and *n*-butyl derivatives are respectively 5, 14, and 26 times as strongly germicidal as resorcinol itself. Details are given for the preparation of these substituted resorcinols and methylresorcinol, the latter compound, however, not being obtained apparently in the pure state. (Cf. J.C.S., May.)—W. G.

*Polysulphide-sulphur.* Wöber. See VII.

*De-aeration of water.* White. See X.

### PATENTS.

*Sterilising liquids; Process of removing germs, ferments, and toxins from and —.* H. Bechhold. G.P. 329,189, 14.3.19. Addn. to 325,505 (J., 1920, 831 A).

THE porous material, e.g., kieselguhr, is coated with a metal, e.g., copper or silver, and with an insoluble compound of another metal, e.g., silver or copper oxide.—L. A. C.

*Antiseptic; Manufacture of a colloidal —.* Chinoïn-Fabr. chem.-pharm. Produkte, A.-G. (v. Kereszty und Wolf). G.P. 329,733, 27.9.18. Conv., 16.5.18.

SUFFICIENT *Magnesia usta*, or a colloidal metallic hydroxide or inorganic acid, e.g., silicic acid, is added to a solution of an alkali, alkaline-earth, or magnesium hypochlorite, to give a mixture which sets within a day to a pasty consistency. The product is as powerful an antiseptic as mercuric chloride for disinfecting the hands and body, but has not its poisonous properties.—L. A. C.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Atropine sulphate from Datura stramonium.* H. W. Rhodehamel and E. H. Stuart. J. Ind. Eng. Chem., 1921, 13, 218—220.

THE following method was used for extracting the alkaloids:—10 tons of the ground plant was percolated with water containing 0.2% of sulphuric acid and 0.5% of formaldehyde; the percolate, in 1500-gal. portions, was then treated with fullers' earth, allowed to settle, the sludge collected, and dried at 50° C. The adsorbed alkaloids were separated from the dried sludge by extraction with alcohol, using lime to obtain the proper alkalinity; the extracts were acidified with acetic acid and concentrated first to 12% and then under reduced pressure to 2% of the original volume. This treatment was sufficient to convert all the hyoscyamine present into its isomeride, atropine. The solution was then rendered ammoniacal, the atropine extracted with ether, the ethereal solution evaporated, and the residual alkaloid dried at 35° C. The dry alkaloid was dissolved in alcohol, the solution neutralised with sulphuric acid, evaporated to a syrup, and the

latter while hot treated with acetone until precipitation almost commenced; on cooling, atropine sulphate was obtained in crystalline form.

—W. P. S.

*Charcoal; Comparative experiments on the adsorptive capacity [for alkaloids, toxins, etc.] of various kinds of —.* F. Horst. Biochem. Zeits., 1921, 113, 99—110.

THE results obtained by the methods of Wiechowski and Joachimoglu (Biochem. Zeits., 1916, 77, 1) for the determination of the adsorptive capacity of charcoal, using solutions of Methylene Blue and of iodine respectively, indicate satisfactorily the adsorptive power of the charcoal for chemically defined poisons (strychnine, neurine), but not for toxins such as diphtheria, tetanus, and ricin toxins.

—S. S. Z.

*Shepherd's purse [Capsellae bursae, Pastoris]. Active constituents of —.* H. Bornuttau and H. Cappenberg. Arch. Pharm., 1921, 259, 33—52.

PHYSIOLOGICAL investigation showed the extracts of the drug to contain a substance which causes very marked depression of the blood pressure and also a product which causes rise in the blood pressure and which is carried down by the precipitate when mercuric chloride is added to the extract. The latter substance is probably tyramine, but it could not be identified with certainty by reason of the small amount of substance available. It is very improbable that histamine is present. Chemical evidence of the presence of an alkaloid could not be obtained with the available material. The isolation of choline and acetylcholine from the liquid extracts is described in detail; the choline bases can also be obtained directly from such preparations by precipitation with alcoholic platonic chloride solution without previous use of lead, mercury, or barium salts, and the following method of evaluating the extract is based on this observation. The extract (1:1, 5 c.c.) is mixed with alcohol (12 c.c.) and treated with alcoholic platonic chloride solution (1%, 20—25 c.c.); the precipitate is filtered off after one or two days, dried, and subsequently extracted on the filter with hot water, the impurities remaining on the filter. The filtrate is evaporated in a tared dish. If necessary, the concentrated filtrate is again filtered or, should it become turbid, is precipitated with 70% alcohol. A good sample of the drug should yield at least 0.2 g. of platinum chloride compounds; the purity of the isolated platinichlorides is controlled by observation of the melting point or by estimation of platinum or nitrogen.—H. W.

*Lobinol, a dermatitant from Rhus diversiloba (Poison oak).* J. B. McNair. J. Amer. Chem. Soc., 1921, 43, 159—164.

LOBINOL, a poisonous, amber-red, oily liquid, has been isolated from the bark of *Rhus diversiloba*. From its chemical behaviour it is probably an unsaturated *o*-dihydric phenol. It is easily darkened, dried, and hardened by means of manganese, barium, or magnesium peroxides, litharge, manganese hydroxide, and potassium bichromate. It hardens at a temperature above 96° C. in the absence of its enzyme or any oxidising agent.

—W. G.

*Siam benzoin. II. Siarensinolic acid.* F. Reintzer. Arch. Pharm., 1921, 259, 1—6.

*d*-SIARENSINOLIC acid (cf. Zinke and Lieb, J., 1918, 520 A) is best prepared by treating the crude benzoin with aqueous sodium hydroxide solution (4—5%) when sodium *d*-siarensinolate remains as a very sparingly soluble precipitate which can be crystallised from alcohol. The free acid has  $[\alpha]_D^{25} = +37.793$  in ethyl alcoholic solution (96%), m.p.



260° C. (Zinke and Lieb, m.p. 274°–275° C.). It does not contain a methoxy group and plays no part in the gradual discoloration suffered by the resin when preserved. Potassium *d*-sarsinolate crystallises in needles which dissolve freely in water and alcohol. The name, *l*-prahangic acid, is proposed for the substance,  $C_{11}H_{10}O_4$ , isolated by Zinke and Lieb by the oxidation of *d*-sarsinolic acid by chromic acid in acetic acid solution.—H. W.

*Siam benzoin*. III. *Properties and constitution of lubanyl benzoate*. F. Reinitzer. Arch. Pharm., 1921, 259, 60–63.

LUBANYL benzoate (J., 1915, 681) crystallises in plates, m.p. 72.8° C., and contains one methoxy group. It is readily oxidised when exposed to warm air and then exhibits a series of colour changes which are precisely analogous to those shown by the crude resin. It readily loses benzoic acid when heated at 120°–140° C., and on further rise of temperature emits the odour of oil of carnations and subsequently of guaiacol. It is optically inactive. The benzoate is very readily hydrolysed either in acid or alkaline solution, but the isolation of lubanol in the pure condition has been impossible up to the present on account of the great susceptibility of the substance, particularly in alkaline solution, to further change. Lubanol is probably identical with or very closely related to coniferyl alcohol.—H. W.

*Oxynitrilase*; *Properties of* —. V. K. Kriebel and W. A. Wieland. J. Amer. Chem. Soc., 1921, 43, 164–175.

The formation of *d*-mandelonitrile by the action of oxynitrilase from peach leaves on benzaldehyde and hydrogen cyanide has a different temperature coefficient from the formation of the inactive nitrile by the spontaneous interaction of the components. At 0° C. the enzyme action is more efficient, but at 35° C. the spontaneous action is the more marked. The ratio of these two actions is, however, very considerably influenced by the hydrogen-ion concentration. (Cf. J.C.S., 1921, i., 283.) —W. G.

*Anæsthesin*; *Derivatives of* —. H. Thoms and K. Ritsert. Ber. deuts. Pharm. Ges., 1921, 31, 65–75.

A NUMBER of derivatives of anæsthesin (ethyl *p*-aminobenzoate) were prepared in which the hydrogen atom of the amino-group was replaced by various positive and negative groups, with the object of determining the effect of such substitution on the anæsthetic properties of the base. In general the physiological action was much reduced by the substitution of the amino hydrogen atoms, and entirely disappeared with the introduction of negative groups. Only in the case of *p*-carbethoxyphenylhydrazine, where an amino hydrogen atom of anæsthesin had been replaced by an amino group, was the anæsthetic action at all comparable with that of anæsthesin itself. Among the substances described are *N*-allyl-*N'*-*p*-carbethoxyphenylthiourea, *p*-carbethoxyphenylhydrazine, *p*-carbethoxyphenylmethylpyrazolone, *p*-carbethoxyphenylurethane, and *p*-nitrobenzoyl-*p*-aminobenzoic acid ethyl ester. (Cf. J.C.S., May.)—G. F. M.

*Anæsthesin and some of its derivatives*; *Anæsthetic action of* —. J. Morgenroth. Ber. deuts. Pharm. Ges., 1921, 31, 76.

COMPARATIVE tests of the anæsthetic action of anæsthesin and certain of its derivatives (cf. *supra*) were made on the cornea of the rabbit. Treatment for 3 mins. with anæsthesin itself rapidly produced anæsthesia lasting 32 mins. *p*-Carbethoxyphenylhydrazine gave almost similar results, whilst *N*-allyl-*N'*-*p*-carbethoxyphenylurea had a scarcely

perceptible action. As regards soluble compounds, carbethoxyphenylhydrazine hydrochloride in 2% solution produced after 3 mins. anæsthesia lasting 9 mins. The insoluble substances thus act more powerfully than their soluble salts.—G. F. M.

*Benzyl esters possessing an antispasmodic action*; *New* —. H. A. Shonle and P. Q. Row. J. Amer. Chem. Soc., 1921, 43, 361–365.

THE benzyl esters of the higher fatty acids may readily be prepared either by the action of benzyl alcohol on the acid chloride or by the action of benzyl chloride on the alkali salt of the fatty acid dissolved in an excess of the hot fatty acid. These esters are tasteless and odourless and have an antispasmodic action. They are more readily hydrolysed by lipase than are the benzyl esters of aromatic acids. Therapeutically the esters of individual acids have nothing to recommend their use over that of the esters of mixtures of fatty acids derived from naturally occurring sources. (Cf. J.C.S., May.)—W. G.

*Benzyl succinate*; *its composition, manufacture, properties, and probable therapeutic uses*. M. Bye. J. Ind. Eng. Chem., 1921, 13, 217–218.

BENZYL succinate is obtained by heating succinic acid with benzyl alcohol; it forms snow-white crystals and is practically non-toxic. The benzyl group constitutes 61.08% of the molecule, and the substance may be used medicinally with advantage in any conditions where the use of benzyl benzoate is indicated.—W. P. S.

*Arsphenamine [salvarsan]; Indirect reduction of 3-amino-4-hydroxyphenylarsonic acid to* —. W. G. Christiansen. J. Amer. Chem. Soc., 1921, 43, 370–375.

3-AMINO-4-HYDROXYPHENYLARSONIC acid is reduced by means of hydriodic acid and sulphur dioxide in hydrochloric acid solution to 3-amino-4-hydroxyphenylarsenious oxide hydrochloride. The excess of sulphur dioxide is removed by aeration and the liquid further reduced by hypophosphorous acid (J., 1921, 97 A) and poured into hydrochloric acid (1:1), the arsphenamine being precipitated as its dihydrochloride in a form readily soluble in water and of low toxicity. If the excess sulphur dioxide is removed by boiling the liquid, the product of the second reduction is arsphenamine polyarsenide. When 3-amino-4-hydroxyphenylarsenious oxide is boiled in hydrochloric acid, *o*-aminophenol is the final product, 3,3'-diamino-4,4'-dihydroxydiphenylarsenious oxide being formed as an intermediate product.—W. G.

*Phenylacetic-*p*-arsonic acid*. G. R. Robertson and J. Stieglitz. J. Amer. Chem. Soc., 1921, 43, 179–181.

PHENYLACETIC-*p*-ARSONIC acid,  $CO_2H.C_6H_4.C_6H_4.AsO_2H$ , prepared from *p*-aminophenylacetic acid by Bart's reaction (cf. J., 1911, 1087), forms white, glistening leaflets, slightly soluble in cold water, very soluble in hot water and in alcohol. When introduced into a heated melting-point bath it melts quietly at about 195° C., but if heated gradually it undergoes change and does not melt below 270° C. (Cf. J.C.S., 1921, i., 281.)—W. G.

*Toluenesulphonamides*; *Separation of *o*- and *p*-* —. W. Herzog and I. Kreidl. Chem.-Zeit., 1921, 45, 231.

A METHOD described by O. Beyer for the separation of *o*- and *p*-toluenesulphonamides by heating the mixture with 10% sulphuric acid, in which it is stated that the *p*-sulphonamide is insoluble, is useless when a large proportion of the *p*-sulphonamide is present, since the whole mixture dissolves. In the case, however, of a mixture containing not



more than 2% of the *p*-sulphonamide, it is possible to separate the latter by dissolving the mixture in hot 10% sulphuric acid; on cooling, the *p*-sulphonamide crystallises out, whilst the greater part of the *o*-sulphonamide remains in solution.—W. P. S.

**Halogenation. XX. Replacement of sulphonic acid groups by halogens.** R. L. Datta and J. C. Bhoomik. *J. Amer. Chem. Soc.*, 1921, 43, 303—315.

THE replacement of sulphonic groups in aromatic compounds by chlorine or bromine (J., 1920, 172 A) takes place generally and may occur with iodine under special conditions. Instead of using free bromine it is better to use a mixture of alkali bromide and bromate, as the bromine is liberated very gradually with simultaneous substitution and the chances of charring and secondary reactions are eliminated altogether. Similarly a mixture of iodide and iodate will in some cases effect a replacement which free iodine itself fails to do. A mixture of chloride and chlorate, however, gives no better results than free chlorine itself. Groups already present in the nucleus exert a great influence on the replacement of sulphonic acid groups by halogens, and certain general rules have been observed. With compounds having one or more hydroxyl groups present in the nucleus the replacement takes place readily at the ordinary temperature, although if there are two hydroxyl groups ortho to one another oxidation often occurs. The presence of an amino group similarly facilitates the replacement, but nitro groups, on the other hand, have a retarding effect. In the case of hydroxycarboxylic acids, the sulphonic group is replaced by halogens, but at the same time the carboxyl group is detached. The presence of halogen atoms in the compound makes the replacement difficult. The presence of alkyl groups also renders the replacement difficult, but the effect is variable. The replacement of the sulphonic group by halogens takes place more easily in the case of a monosulphonic acid than in the case of a di- or a trisulphonic acid. (Cf. J.C.S., May.)—W. G.

**Epichlorohydrin; Action of — on disodium phosphate in aqueous solution and the stability of a monoglyceromonomorphoric di-ester.** O. Bailly. *Comptes rend.*, 1921, 172, 689—691.

WHEN epichlorohydrin and disodium phosphate are mixed together in aqueous solution in equimolecular proportions and the solution is allowed to stand, the amount of phosphorus precipitable by magnesia mixture steadily diminishes and there is a progressive formation of sodium chloride. Under these conditions a mixture of a mixed glycidochloroglycerophosphoric ester and a monoglyceromonomorphoric di-ester,  $\text{NaO} \cdot \text{PO}(\text{O} \cdot \text{CH}_2)_2 \cdot \text{CH}_2\text{OH}$ , probably results, but if the solution is boiled for 2 hrs., the di-ester is almost the sole product. It is remarkable that this di-ester should be stable in boiling solution.—W. G.

**Cyanic acid and urea; Synthesis of — by oxidation in ammoniacal solution of alcohols, phenols, and aldehydes.** R. Fosse and G. Laude. *Comptes rend.*, 1921, 172, 684—686.

METHYL, ethyl, and butyl alcohols, phenol, *o*-cresol,  $\alpha$ - and  $\beta$ -naphthols, catechol, resorcinol, acetaldehyde, propaldehyde, and butaldehyde, all yielded cyanic acid on oxidation in ammoniacal solution with potassium permanganate alone or in the presence of ammonium sulphate, copper carbonate, or copper powder. The cyanic acid was converted into urea by treatment with ammonium chloride.—W. G.

**Triacetin; Successive stages of hydrolysis of —.** E. Yamasaki. *Sci. Rep. Tohoku Imp. Univ.*, 1920, 9, 451—471.

IF the rate of hydrolysis of each of the three ester radicles in triacetin is equal and the reaction is in each case unimolecular, it follows that, if  $k_1$  is the constant for the reaction  $\text{C}_2\text{H}_5(\text{O} \cdot \text{C}_2\text{H}_5\text{O})_2 + \text{C}_2\text{H}_5(\text{OH})(\text{O} \cdot \text{C}_2\text{H}_5\text{O})_2 + \text{CH}_3\text{CO}_2\text{H}$ ,  $k_2$  for the reaction  $\text{C}_2\text{H}_5(\text{O} \cdot \text{C}_2\text{H}_5\text{O})(\text{OH}) + \text{C}_2\text{H}_5(\text{OH})(\text{O} \cdot \text{C}_2\text{H}_5\text{O}) + \text{CH}_3\text{CO}_2\text{H}$ , and  $k_3$  for the reaction  $\text{C}_2\text{H}_5(\text{OH})(\text{O} \cdot \text{C}_2\text{H}_5\text{O}) + \text{C}_2\text{H}_5(\text{OH}) + \text{CH}_3\text{CO}_2\text{H}$ , the relation between these constants is such that  $k_1/3 = k_2/2 = k_3 = k$ . This conclusion was verified experimentally, the velocity constants being determined graphically from the hydrolysis curves of triacetin, diacetin, and monoacetin. The experiments were carried out at 35° C. and the curves obtained by plotting the concentration of acetic acid against time. Knowing the value of  $k$ , the concentrations of mono-, di-, and triacetin at any time can be calculated. The velocity of hydrolysis is approximately doubled for each 10° C. rise in temperature.—E. H. R.

**Amines of secondary alcohols; Preparation of —.** A. Mailhe. *Comptes rend.*, 1921, 172, 692—694.

THE hydrogenation of ketazines of symmetrical ketones (cf. *Comptes rend.*, 1920, 170, 1265) has been successfully extended to the hydrogenation of the ketazines of unsymmetrical ketones. The product is in all cases a mixture of primary and secondary amines of secondary alcohols. Numerous examples are given. (Cf. J.C.S., May.)—W. G.

**Tetrachloroethane and trichloroethylene; Preparation of — from acetylene and chlorine.** S. Igi. *Kogyō Kwagaku Zasshi (J. Chem. Ind. Japan)*, 1920, 23, 1217—1237.

TETRACHLOROETHANE is prepared by passing alternately acetylene at 60°—80° C. and chlorine at 80°—100° C. into antimony pentachloride. 100 g. of antimony chlorido suffices for the preparation of 150—180 g. of tetrachloroethane; the recovery of the catalyst is difficult and unprofitable. The rate of absorption of the acetylene and chlorine by the pentachloride is at first high, but diminishes rapidly; the effect of impurities is not large. Trichloroethylene is prepared by heating a mixture of tetrachloroethane and milk of lime, a yield of 84% of the theoretical value being obtained. Some excess of lime is advantageous, e.g., 60 g. of slaked lime and 50 g. of water for 100 g. of tetrachloroethane.—K. K.

**Formaldehyde; Adsorption of — by animal charcoal.** W. Moeller. *Kolloid-Zeits.*, 1921, 28, 127—132.

FORMALDEHYDE is strongly adsorbed by animal charcoal both from acid and neutral solutions. In acid solution the adsorption depends on the volume of solution, but this is not the case in neutral solutions. Equilibrium is set up immediately in neutral solutions, but in acid solutions is not complete even after eight days.—J. F. S.

**Oxalic acid; Formation of — from sulphite-cellulose waste liquors and from lignin.** E. Heuser, H. Roeseb, and L. Gunkel. *Cellulosechem.*, 1921, 2, 13—19.

LIGNIN free from carbohydrates produced no oxalic acid on fusion with potassium hydroxide at temperatures up to 270° C.; sulphite waste liquor under similar conditions often yield oxalic acid owing to the presence of cellulose and other carbohydrates in them. Oxidation of lignin with fuming nitric acid gave yields of oxalic acid up to 25% (20% actually isolated); addition of small quantities of ferrous or mercurous sulphate or ammonium vanadate reduced the yield, and the use of mixture

of nitric and sulphuric acids produced no oxalic acid at all, doubtless owing to the destructive action of the sulphuric acid. On heating 3 g. of lignin with 60 g. of dilute nitric acid (25%) for 4 hrs. at 80°–90° C., a clear solution was obtained from which 0.13 g. of oxalic acid was isolated; the yield was increased to over 17% by addition of 0.01 g. of ferrous or mercurous sulphate, but larger amounts of these catalysts acted less favourably.—J. H. L.

*Mustard oil [allyl isothiocyanate]; Cause and composition of the insoluble deposits in —.* H. Kunz-Krause. Arch. Pharm., 1921, 259, 16–33.

NATURAL and synthetic mustard oil becomes discoloured when preserved and slowly deposits a dark orange to lemon yellow precipitate. The latter contains colourless quadratic prisms and small quantities of hexagonal plates, cubes, and slender prismatic needles. A portion of the deposit is soluble in water and consists principally of allylamine sulphate possibly admixed with traces of ammonium sulphate. The chief constituent of the portion insoluble in water is pseudothiocyanogen,  $C_3H_5S_2$ , whilst isopercythiocyanic acid,  $C_3H_5S_2N_2$ , and allyl-substituted ureas are most probably also present. The formation of the substances is traced to the conjoint action of air, moisture, and light on the oil, which is best preserved in small, well-filled, dry bottles of green glass. (Of. J.C.S., May.)

—H. W.

*Wormseed oil; Reaction of American —.* A. Langer. Pharm. Zeit., 1921, 66, 191.

OIL of American wormseed (*Chenopodium ambrosioides*) yields a red-yellow resin when heated at about 175° C. with phenolphthalein in the presence of toluene; 10 g. of the oil requires about 4 g. of phenolphthalein. After the product has been steam distilled to remove terpenes and toluene, any excess of phenolphthalein may be removed by dissolving the resin in warm toluene. The resin is soluble in chloroform, alcohol, ether, and alkali solutions and is precipitated by acids from the latter solutions as a yellow powder which melts below 100° C. to give the red resin.—W. P. S.

*Thymol and carvacrol; Urethanes of —.* D. C. L. Sherck. Amer. J. Pharm., 1921, 93, 115–125.

THE urethanes mentioned below were prepared by simply heating a mixture of thymol or carvacrol and the phenolic isocyanate together with a paraffin hydrocarbon of b.p. 170°–200° C. for about 1 hr.; the urethanes crystallised from the mixture on cooling. Thymol phenyl urethane, m.p. 106.5°–107° C., yield 71%; carvacrol phenyl urethane, m.p. 138° C., yield 86%; hydrothymoquinone phenyl diurethane, m.p. 229°–230° C., yield 84%; thymol  $\alpha$ -naphthyl urethane, m.p. 156°–157° C., yield 43%; carvacrol  $\alpha$ -naphthyl urethane, m.p. 117°–119° C., yield 36%. Dibenzoylhydrothymoquinone, m.p. 141°–142° C., yield 75%, was obtained by dissolving hydrothymoquinone in a slight excess of 15% sodium hydroxide solution and then adding benzoyl chloride.—W. P. S.

*Borneol and its alkyl derivatives; Determination of — by acetylation.* F. Martin. J. Pharm. Chim., 1921, 23, 168–171.

BORNEOL is acetylated completely when heated at 145°–150° C. for 3 hrs. with three times its weight of acetic anhydride and a small quantity of fused sodium acetate. After the acetylated product has been washed with water and 5% sodium carbonate solution, and dried over anhydrous sodium sulphate, the quantity of borneol present is found by determining the saponif. value of the acetylated product; in the calculation allowance is made for

the increase of molecular weight (by 42 units) due to the acetylation.—W. P. S.

*Oligodynamic action of the heavy metals and of the salts of the heavy metals; So-called —.* W. Falta and M. Richter-Quittner. Biochem. Zeits., 1921, 115, 39–41.

THE oligodynamic action of various metals was tested in the following way:—a test-tube was filled with water containing the respective metals and was allowed to stand for eight days. The metal and the water were then removed, the tube rinsed out with distilled water, and its oligodynamic action tested on different solutions. It was found that guaiacol, benzidine, resorcinol, and homogentisinic acid were oxidised when kept in test-tubes prepared in the above way. Potassium permanganate was decolorised with the formation of manganese dioxide. Methylene Blue, Indigo Blue, and sodium indigosulphonate were decolorised and the leucobase of Malachite Green was oxidised. Protein solutions were coagulated through the oligodynamic action of the metals. This oligodynamic action was shown by the following metals which are arranged in the order of their activity:—copper, mercury, silver, lead, tin, aluminium, iron, magnesium, and platinum.—S. S. Z.

*Otoba butter.* Baughman and others. See XII.

*Methyl anthranilate.* Power. See XIXa.

*Arsenic in organic compounds.* Robertson. See XXIII.

#### PATENTS.

*Tea plants; Treatment of the waste cut from growing — and the obtaining of caffeine therefrom.* R. L. Datta. E.P. 159,097, 1.3.20.

THE leaves and stems cut from growing tea plants are dried, ground, and extracted with water. The aqueous extract is treated with lime, and the calcium tannate precipitated is separated by filtration. Crude caffeine crystallises from the solution after concentration, and is purified by solution in chloroform, filtration, evaporation of the solvent, and recrystallisation of the product from hot water.

—L. A. C.

*Copper-tannin-albumin compound; Preparation of a —.* P. Beiersdorf und Co. G.P. 325,957, 24.6.19.

A COMPOUND in which the therapeutic properties of tannin are improved by the presence of copper, is prepared by heating a mixture of an alkaline casein solution, a copper salt, and a tannin solution. The product, which contains 5.42% H, 48.85% C, 7.45% N, and 4.95% Cu, is an odourless, brown powder with a faint metallic taste.

—L. A. C.

*Salts corresponding to the salts contained in the human blood; Manufacture of a mixture of dry — soluble in water to a clear solution.* Sächsisches Serumwerk und Inst. für Bakteriotherapie, G.m.b.H. G.P. 329,309, 7.10.19.

A SALT mixture from which no calcium salts separate on solution in water is prepared by adding powdered calcium chloride, of which each particle is coated with a layer of a colloid, such as gelatin, which swells on addition of water, to a mixture of blood serum salts.—L. A. C.

*Mercury-casein ointment or emulsion; Manufacture of a —.* C. Biedermann. G.P. 329,429, 29.5.18.

MERCURY is worked up to a paste with neutral, freshly precipitated casein and water to yield a non-irritant ointment.—L. A. C.

*Salves and emulsions; Bases for* — capable of holding a large proportion of water. I. Lifschütz. G.P. 329,605, 11.5.19. Addn. to 324,012 (J., 1920, 833 A).

ANIMAL fats prepared from blood or brains containing a large proportion of metacholesterol are employed instead of pure metacholesterol, as described in the chief patent.—L. A. C.

*Salves; Manufacture of adsorptive* —. Elektro-Osmose A.-G. (Graf Schwerin Ges.). G.P. 329,672, 24.2.16.

COLLOIDAL silicic acid containing, e.g., 12% of silicic acid, is mixed with 5% of glycerin, 10% of paraffin or vaseline, 25% of Peru balsam, or with other similar materials.—L. A. C.

*Furfuralacrolein; Preparation of* —. W. König and K. Hey. G.P. 330,358, 12.11.19.

FURFURALACROLEIN, m.p. 54° C., which is suitable for use as a flavouring essence or perfume, is prepared by the gradual addition of acetaldehyde to an alkaline solution of furfural at a low temperature, e.g., 0° C.—L. A. C.

*Methyl bromide; Production of* —. G. Schroeter. G.P. 330,642, 25.11.17.

A MIXTURE of methane, or gases containing the same, and bromine is led, above 200° C., over a catalyst such as iron, copper, nickel, or cobalt, or other metals having more than one valency, or mixtures of the same. If methane is bubbled through a depth of 5–6 cm. of liquid bromine and led, at a velocity of 2 litres per min., through tubes of 20 mm. diam. containing iron powder and heated for a length of 250 mm. to 250°–300° C., the product contains 50% of unchanged methane; if the proportion of bromine in the gas is increased, methylene dibromide and tribromomethane are formed in addition to methyl bromide.—L. A. C.

*Cinchona alkaloids; Preparation of compounds containing selenium from hydrogenated* —. Ver. Chininfabr. Zimmer und Co., G.m.b.H. G.P. 331,145, 30.11.17.

COMPOUNDS of therapeutic value are prepared by the action of selenium dioxide on hydrogenated cinchona alkaloids, or their derivatives, in the presence of concentrated sulphuric acid. The product is diluted with water, and boiled. Selenohydroquinine, prepared from hydroquinine sulphate or hydroquinine sulphuric acid ester, forms bright yellow needles from alcohol, m.p. above 235° C.; selenoethylhydrocupreine forms yellow needles, m.p. 233°–234° C., and selenohydrocupreine forms bright orange needles, m.p. above 235° C., soluble in sodium hydroxide solution.—L. A. C.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Reflection and density meter for photographic papers*. K. Kieser. Phot. Korr., 1920, 17, 287–289.

THE paper to be tested is illuminated at 45° and viewed through a polarisation photometer, at 45° for reflection measurements and at 90° for density measurements.—B. V. S.

*Eder-Hecht grey-wedge photometer*. J. M. Eder. Phot. Korr., 1920, 57, 304–307.

SEVERAL minor improvements have been made in the instrument (J., 1920, 803 A). Particulars are given, with curves, of the spectral absorptions of the four colour-filters, red, yellow, light green, and blue, which are combined with the grey wedge.—B. V. S.

*Safranine-quinol, a cheap and rapid developer*. Lüppo-Cramer. Der Photograph, 1921, 65–66.

THE addition of Phenosafranine to an ordinary caustic alkali quinol developer, up to a strength of 1:20,000, produces a developer resembling metol-quinol and nearly, though not quite, as rapid in action. A further increase in the proportion of Phenosafranine has practically no effect on the speed. Where the most rapid development is not essential this developer is recommended on account of its cheapness, while it has the additional advantage of allowing development in bright light.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

*Nitrous acid; Determination of* — in mixed and waste acids. H. Toussaint. Z. angew. Chem., 1921, 34, 102.

FOR the estimation a Woulff bottle with three necks is used, through the outer ones of which pass a funnel, and an inlet tube for carbon dioxide, respectively. 700 c.c. of air-free, distilled water is placed in the bottle, and the air is displaced by carbon dioxide. A suitable amount of the acid to be tested is run in through the middle neck from a burette, after which a glass tube, 10 cm. long, is fixed in the neck by a collar of rubber tubing, so that its lower end is flush with the top of the bottle; the carbon dioxide tube is next pushed down to the bottom of the bottle. Potassium iodide solution is run in through the funnel and the solution titrated with  $N/2$  or  $N/10$  thiosulphate, introduced through the middle neck, together with starch solution if desired, the titration being conducted in a slow current of carbon dioxide. If the acids contain a considerable amount of ferric oxide, this must first be removed by treatment with sodium hydroxide, free from nitrate, after which the filtrate is acidified with pure sulphuric acid.—W. J. W.

### PATENTS.

*Smokeless powders; Manufacture of* —. Westfälisch-Anhaltische-Sprengstoff-A.-G. G.P. 329,775, 15.7.13.

ADDITION of di-substituted urethanes, especially methylphenylurethane, to mixtures of nitroglycerin and nitrocellulose containing not more than 30% of nitroglycerin, enables the material to be pressed at temperatures below 82° C. Volatilisation of the nitroglycerin is thereby reduced, and a stable explosive is produced.—W. J. W.

*Matches; Manufacture of damp-proof* —. R. Dubrisay. E.P. 145,798, 2.7.20. Conv., 3.7.19.

SEE U.S.P. 1,363,095 of 1920; J., 1921, 130 A.

## XXIII.—ANALYSIS.

*Analysis; Use of spot reactions in qualitative* —. F. Feigl and R. Stern. Z. anal. Chem., 1921, 60, 1–43.

SPOT reactions, usually made on filter paper, are shown to be applicable to the identification and detection of a large number of substances when these occur either alone or in mixtures. The reactions of aluminium, uranium, and chromium with alizarin dyes (formation of lakes) are very sensitive, as is also the reaction of manganese with benzidine.—W. P. S.

*Analysis by means of reducing flames; Principles of* —: detection of traces of manganese in the presence of iron or other substances. J. Meunier. Comptes rend., 1921, 172, 678–681.

WHEN an oxide or a salt, introduced into a flame, undergoes reduction, the reaction is manifested by

a line spectrum. If the oxide is not reduced the spectrum of the metal is not produced, but only a continuous spectrum of incandescence. It is necessary in many cases to use a hydrogen flame, as its reducing action is more energetic than that of a gas flame. In this method manganese is characterised by the triplet 4034.7, 4033.2, 4030.9 A. units, which is of extraordinary sensitiveness.—W. G.

*Analysis; Physico-chemical volumetric* — R. Dubrisay. *Ann. Falsif.*, 1921, 14, 9–18.

PHYSICAL methods for determining the end-point of titrations are discussed, those dealt with including electrical conductivity, surface tension, and cryoscopic measurements.—W. P. S.

*Filter paper; Adsorption of lead and copper by* — [in analysis]. I. M. Kolthoff. *Pharm. Weekblad*, 1921, 58, 152–159.

THE adsorption of lead from its solutions by filter paper is proportional to the alkalinity of the filter ash. With a high alkalinity nearly all the lead may be removed from a solution, whereas with a negligible alkalinity no appreciable adsorption takes place. On the other hand, the adsorption of copper from solution bears no relation to the alkalinity of the filter ash. From neutral solutions no adsorption takes place; from solutions in ammonia the adsorption is high, and it is greater with low concentrations than with higher ones. (*Cf. J.C.S.*, April.)—W. J. W.

*Adsorption; Importance of* — in analytical chemistry. VII. *Adsorption of silver, mercury, and other metals*. I. M. Kolthoff. *Pharm. Weekblad*, 1921, 58, 233–241.

THE fixation of heavy metals is due to a chemical reaction with the ash of the cellulose, which is inhibited by acids. (*Cf. J.C.S.*, April.)—S. I. L.

*Microchemical analysis with reagents sensitised by saturation*. E. Ludwig. *Bul. Soc. Chim. România*, 1920, 2, 23–32.

THE reagent to be used is saturated with a salt containing the ion which is to be detected, the formation of a precipitate being observed under a microscope. The reagents recommended are  $N/2$  silver nitrate saturated with sodium sulphate for the detection of sulphates; potassium pyroantimonate solution saturated with sodium chloride for the detection of sodium, and a solution of platinum chloride saturated with potassium chloride for the detection of potassium.—W. G.

*Microchemical analysis with reagents sensitised by saturation*. E. Ludwig and D. Butescu. *Bul. Soc. Chim., România*, 1920, 2, 32–35. (*Cf. preceding abstract.*)

THE reagents recommended are  $N/2$  silver nitrate treated with sodium acetate, until a precipitate just appears, for the detection of acetates; a solution of uranyl acetate saturated with a sodium salt for the detection of sodium. For the identification of borax a few particles are added to a drop of  $N/2$  silver nitrate. Very small crystals soon begin to appear, and on warming the slide the undissolved particles become black.—W. G.

*Sodium acetate method for the separation of the divalent metals from trivalent metals in the ammonium sulphide group*. T. Sabalitschka. *Ber. deuts. Pharm. Ges.*, 1921, 31, 36–37.

THE sodium acetate method described by Macri (*J.*, 1920, 804 A) for the separation of iron, chromium, and aluminium from zinc, manganese, etc., is useless in the presence of chromium, as this metal is not precipitated at all when alone, and only partially in presence of iron and aluminium. —G. F. M.

*Potash; Centrifugal method for determining* — [in molasses, etc.]. E. Sherrill. *J. Ind. Eng. Chem.*, 1921, 13, 227–228.

FIVE c.c. of a solution of the sample, containing about 1%  $K_2O$ , is added to 17 c.c. of sodium cobaltinitrite solution, contained in a centrifuge tube; a similar mixture is prepared with 5 c.c. of 1.583% potassium chloride solution (=1%  $K_2O$ ) and both mixtures are centrifuged for 1 min. at 1000 revs. per min. The tubes are then tapped with the finger to level the surface of the precipitate, again centrifuged for 15 secs., and the volumes of the precipitates noted. In the case of molasses, about 26 g. is dissolved in water, rendered alkaline with sodium hydroxide, acidified with acetic acid, diluted to 100 c.c., and 5 c.c. of this solution used for the determination. The sodium cobaltinitrite solution is prepared by adding 450 g. of sodium nitrite dissolved in 800 c.c. of water to 250 g. of cobalt acetate dissolved in 800 c.c. of water, and diluting the mixture to 2000 c.c.; for use, 100 c.c. is mixed with 65 c.c. of water and 5 c.c. of glacial acetic acid. The acidified reagent does not keep well.—W. P. S.

*Manganese; Interference of phosphates in the detection of* — and its avoidance. T. Sabalitschka and H. Niesemann. *Ber. deuts. Pharm. Ges.*, 1921, 31, 30–36. (*Cf. J.*, 1921, 64 A.)

THE failure to detect manganese in presence of an excess of barium or strontium phosphate when the separation of phosphoric acid is conducted according to Schmidt's method is obviated by the following procedure:—The precipitate obtained with ammonium sulphide is dissolved in 5% hydrochloric acid and filtered from insoluble nickel and cobalt sulphides. The solution is evaporated with 2–3 g. of concentrated sulphuric acid and a like quantity of saturated potassium sulphate solution until white fumes are evolved, any ferrous iron being previously oxidised by nitric acid. The mixed sulphates obtained are extracted with alcohol to remove sulphuric and phosphoric acids (small quantities of manganese and zinc sulphates and most of the chromium also pass into solution). The residue insoluble in alcohol is extracted with dilute hydrochloric acid and the insoluble sulphates of barium, strontium, and calcium filtered off. From the filtrate, iron, aluminium, and chromium are precipitated with ammonia, and then the sulphides of zinc and manganese with ammonium sulphide. The final filtrate contains calcium and magnesium. —G. F. M.

*Iron; Volumetric determination of* — by titanous salts. W. M. Thornton, jun., and J. E. Chapman. *J. Amer. Chem. Soc.*, 1921, 43, 91–102.

KNECHT and Hibbert's method (*J.*, 1903, 232, 762) of estimating ferric iron by titration with a solution of a titanous salt using ammonium thiocyanate as indicator yields trustworthy results under the following conditions. The titanium solution (either chloride or sulphate) must be preserved under hydrogen both in the storage bottle and in the burette, and the titration must be carried out in an atmosphere of carbon dioxide and at a temperature not above 30° C. Not more than 500 c.c. of solution may be titrated and 10 c.c. of 10% solution of ammonium thiocyanate should be added. In presence of hydrofluoric acid the indicator gives only a yellow coloration, but if an excess of boric acid is added the red colour is produced and a perfectly sharp end point is obtainable. The titanous solution is standardised with ferrous ammonium sulphate which is exactly oxidised by potassium permanganate and then reduced with the titanous solution. Standard titanous solutions preserved as described maintain their titre indefinitely.—J. F. S.

*Copper, antimony, and tin; Electro-analytical separation of —.* F. Foerster and D. Aanensen. *Z. Elektrochem.*, 1921, 27, 10—16.

THE solution containing copper, tin, and antimony is evaporated to dryness, treated with 5 c.c. of nitric acid (sp. gr. 1.4), 5 c.c. of 50% tartaric acid, and sufficient hydrogen peroxide to oxidise the tin, and made up to 40 c.c. with water. The solution is electrolysed, with thorough cooling, for 1 hr. with a current of 1.5 amp. in a Frary apparatus with a 5 amp. coil current. The copper deposit is removed with nitric acid and tartaric acid, and the copper re-deposited in the same manner. Since the first deposit contains no tin but only antimony as impurity, the two solutions after the second deposition of copper are not mixed, but each is neutralised with sodium hydroxide, treated with 50 c.c. of 10% oxalic acid, and treated hot with hydrogen sulphide. The two antimony precipitates are united, dissolved in alkali sulphide, and 30 c.c. of 30% potassium cyanide added. The solution is then electrolysed at 70° C. with 0.6 amp., using a Winkler net electrode. The deposition requires 2 hrs. for 0.1 g. of antimony, and the deposit is 1.8% heavier than the amount of antimony present. The solution containing the tin is concentrated, neutralised with 50% potassium hydroxide, made alkaline with 2 c.c. of 50% potassium hydroxide, treated with 1 c.c. of 3% hydrogen peroxide, boiled, and diluted to 150 c.c. The tin is deposited in 3½ hrs. at 80°—85° C. on a Winkler electrode with a current of 5 amp.; during the electrolysis 3 c.c. of 50% potassium hydroxide is added along with water to make good the loss by evaporation.—J. F. S.

*Antimony and tin; Separation of — by hydrogen sulphide in hydrochloric acid solution.* G. Luff. *Chem.-Zeit.*, 1921, 45, 229—231.

THE different behaviour of antimony and tin sulphides in hydrochloric acid solution of varying concentration and at different temperatures was investigated. Precipitation of antimony sulphide begins at 80° C. when a solution of the two metals containing 30 c.c. of concentrated hydrochloric acid per 100 c.c. is treated with hydrogen sulphide; the tin sulphide remains in solution even at 25° C.

—W. P. S.

*Tin and antimony; Separation of — in hydrochloric acid solution by means of hydrogen sulphide.* G. Luff. *Chem.-Zeit.*, 1921, 45, 249—251, 254—255, 274. (Cf. *supra*.)

IF hydrogen sulphide is passed into boiling solutions of antimony and tin salts, with addition of concentrated hydrochloric acid, increasing concentrations of the latter lower the temperature at which precipitation takes place on cooling. With an addition of 8, 14, and 30 c.c., respectively, to 100 c.c. of solution, the temperatures of precipitation were 102°, 102°, and 95° C. for antimony trisulphide; 102°, 100°, and 80° C. for the pentasulphide; and 90°—95°, 75°—80°, and 25° C. for stannic sulphide. Ammonium chloride still further lowers the temperature at which the sulphides separate; with 36 g. per 100 c.c. of solution the trisulphide and pentasulphide are precipitated at 78° and 60° C. respectively, whilst even at normal temperature no precipitation of the stannic sulphide occurs till after standing. The effect on the separation of the sulphides of varying concentrations of hydrochloric acid, in presence of a fixed amount (16.5%) of ammonium chloride, has been investigated. The cleanest separation takes place with 14 c.c. of hydrochloric acid (sp. gr. 1.193) per 100 c.c. of solution; the lowest effective amount is 8 c.c. per 100 c.c., and the maximum amount appears to be 35 c.c. per 65 c.c. of solution. For separating antimonious salts, Vortmann and Metzger's

method (19.4 c.c. of hydrochloric acid) as well as modifications with 8 and 14 c.c., are all satisfactory. The Panajotow-Prim method (30 c.c.) gives sufficiently accurate results; when the ammonium chloride exceeds 16.5 g., however, the solution must be cooled in ice, and the introduction of hydrogen sulphide checked at 20° C., filtration being conducted at 5° C.—W. J. W.

*Arsenic and phosphoric acids; Determination of — in presence of large quantities of salts.* L. Desboursdeux. *Bull. Sci. Pharmacol.*, 1920, 27, 225—240, 300—313, 363—372, 424—435. *Chem. Zentr.*, 1921, 92, II, 475.

THE ordinary methods for the determination of arsenic and phosphoric acids are not satisfactory when applied to solutions containing large quantities of alkali salts, such as are obtained by decomposition of organic substances, low in arsenic and phosphorus, with alkali nitrate and carbonate or with persulphate. Silver arsenate and phosphate are practically insoluble in a neutral solution containing ammonium salts and an excess of silver nitrate. The following procedure is, therefore, recommended. The solution, if not acid, is treated with 5—10 c.c. of strong nitric acid, then with sufficient silver nitrate solution to precipitate all the chloride, phosphate, and arsenate present and leave an excess of at least 2 g. of silver nitrate per litre of solution. The liquid is then carefully and exactly neutralised with ammonia, allowed to stand for ½ hr., and, if necessary, again brought to neutrality. Tests must be made on a spot plate, as indicators are precipitated by silver salts. The precipitate is filtered off, washed four times with 25 c.c. of 0.2% silver nitrate solution, and digested with a slight excess of a solution containing 40 c.c. of strong nitric acid and 4 g. of barium nitrate per litre to dissolve out the silver phosphate and arsenate and precipitate any adsorbed sulphate. After standing for 1 hr. the insoluble material is filtered off and washed with a solution containing 20 g. of strong nitric acid, 0.5 g. of barium nitrate, and 4 g. of silver nitrate per l.; the filtrate is exactly neutralised with ammonia as before, the precipitate collected in an alundum crucible, washed with 50—100 c.c. of 0.2% silver nitrate solution, then with distilled water till free from nitrates, dried at 150° C., then heated at 400°—500° C. to remove any ammonium nitrate, and weighed. Silica must be removed, if present, by evaporation of the solution of the first silver precipitate to dryness. If a large amount of chloride or any chromate is present the original solution is treated with caustic soda, boiled to expel any ammonia, cooled, and the arsenic and phosphoric acids precipitated by addition of a salt of an alkaline-earth metal. The precipitate is dissolved in nitric acid and precipitated with silver nitrate as described above.—A. R. P.

*Arsenic; Detection of —.* L. W. Winkler. *Pharm. Zentralh.*, 1921, 62, 125—128.

THREE tests are described for the detection of small quantities of arsenic, namely, Bettendorf's test (use of stannous chloride dissolved in hydrochloric acid), a test in which arsine is detected by means of Nessler reagent, and a modification of the Gutzeit test, in which arsine is detected by the stain it gives on paper moistened with gold chloride solution. The Bettendorf test can be applied in the presence of sulphuric acid; in the case of dark-coloured solutions it is necessary to treat these with zinc and sulphuric acid, pass the evolved gases through hydrochloric acid, and test this for arsenic. Antimony, sulphur, and phosphorus compounds interfere with the Nessler reagent test, as they also produce precipitates.—W. P. S.

**Arsenic; Determination of** — in organic compounds. G. R. Robertson. J. Amer. Chem. Soc., 1921, 43, 182–185.

The compound is oxidised by digestion with a mixture of nitric and sulphuric acids (*cf.* Morgan, Organic compounds of arsenic and antimony, 1918, p. 349), the liquid is freed from nitrous compounds by means of ammonium sulphate, and the arsenic estimated by the iodometric method of Gooch, Browning, and Morris (Amer. J. Sci., 1900, 10, 151). The results are quite comparable with those obtained by the Carius method.—W. G.

**Tungsten; Analytical chemistry of** — G. Fiorantino. Giorn. Chim. Ind. Appl., 1921, 3, 56–58.

For volumetric estimation an ammoniacal solution of an alkali tungstate, which should not contain excess of alkali salts, or of hydrated tungstic acid, containing about 0.25 g.  $\text{WO}_3$ , is diluted to about 200 c.c., acidified with acetic acid, then rendered alkaline to the extent of one or two drops of ammonia solution, and boiled for a few minutes until a litmus paper in the solution becomes wine-red. To the boiling liquid, a measured volume, in excess, of standard lead acetate solution (32.6393 g. of the crystallised salt and 2 c.c. of acetic acid per 2 l.) is added, with continual stirring, boiling being continued until the flocculent precipitate becomes pulverulent or crystalline. Standard ammonium molybdate (7.485 g. of crystallised salt per l.) is then run into the boiling solution until a drop of the liquid gives a yellow coloration with a drop of a fresh solution of 0.1 g. of tannin in 10 c.c. of water. A blank test is carried out with 200 c.c. of water to ascertain the volume (usually 0.3 c.c.) of the molybdate solution necessary to yield a distinct yellow coloration with the tannin solution. Multiplication by 1.202 of the amount of lead precipitated by the tungstic acid gives the corresponding amount of  $\text{WO}_3$ . The reactions on which the method is based are  $\text{H}_2\text{WO}_4 + \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O} = \text{PbWO}_4 + 2\text{C}_2\text{H}_3\text{O}_2 + 3\text{H}_2\text{O}$ , and  $\text{Mo}_2\text{O}_7(\text{NH}_4)_4 \cdot 4\text{H}_2\text{O} + 7\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O} = 7\text{PbMoO}_4 + 6\text{NH}_4\text{C}_2\text{H}_3\text{O}_2 + 8\text{C}_2\text{H}_3\text{O}_2 + 21\text{H}_2\text{O}$ . With an ore or concentrate, 1.5 g. is finely powdered and heated to 60° C. for 1 hr. with 100 c.c. of concentrated hydrochloric acid in a covered beaker. The beaker is then heated more strongly on a sand-bath, stirring being continuous for at least 30 mins. and afterwards occasional. When the liquid is reduced to about one-half its original volume 20 c.c. of nitric acid is added, and the liquid evaporated to about 25 c.c., 5 c.c. of nitric acid being then added and the liquid evaporated to 15–20 c.c. and mixed with about 180 c.c. of boiling water. After some hours the liquid is filtered and the precipitate washed by decantation with 1% nitric acid. If highly accurate results are not required the filter is returned to the beaker and the contents heated gently with 8–10 c.c. of concentrated ammonia solution and 25 c.c. of water until all the tungstic acid is dissolved; the undissolved residue should contain no black particles of undecomposed ore and should be whitish or but slightly coloured. With a poor ore the liquid containing the filter-paper is washed into a tall 350 c.c. beaker, the volume being made up to about 200 c.c. and the tungstic acid determined as above; with a rich ore the liquid is made up to a definite volume and an aliquot part taken so that the amount of  $\text{WO}_3$  is about 0.25 g. If very exact results are required the filter is washed with the mixture of 8–10 c.c. of ammonia and 25 c.c. of water, the liquid being collected in the beaker containing the bulk of the precipitate. This beaker is then heated gently until all the tungstic acid is dissolved, the solution being filtered through the same filter, and the latter washed with very dilute ammonia solution. The solution is dealt with as described above, while the filter containing the

residue is dried and calcined in a porcelain crucible at a moderate temperature, any tungstic acid it contains being estimated by the cinchonine method (see below); the latter also admits of the estimation of the tin, which will be contained in the residue. The acid liquid from the original attack of the ore and from the washing may also be examined by concentration and precipitation with cinchonine, since if too little acid remains when the boiling water is added, part of the tungstic acid may be hydrolysed and pass into solution. The cinchonine gravimetric method is carried out as follows: About 1 g. of pure sodium hydroxide is fused with a trace of wood charcoal in a small iron crucible and, when the water has been expelled, allowed to cool. As soon as solidification occurs 0.5–1 g. of the tungsten ore is added, together with a little wood charcoal, the crucible is covered with an iron lid and heated gradually to dull redness, which is maintained for 8–10 mins. The fused mass is poured into a clean, dry iron or nickel dish floating on water, the crucible being freed from adherent material by gentle warming with water in a porcelain dish, into which also the solidified mass from the metal dish is introduced. The whole mass is dissolved by boiling, finally with addition of 4–5 g. of solid ammonium carbonate, and the liquid filtered, the filter being washed with water rendered slightly alkaline with sodium hydroxide. The filtrate is heated and made strongly acid by gradual addition of 30 c.c. of concentrated hydrochloric acid, boiled for a few minutes, mixed well with 8–10 c.c. of a solution of 30 g. of cinchonine in 50 c.c. of concentrated hydrochloric acid and 250 c.c. of water, allowed to stand for some hours and filtered through a filter containing a little pulped filter-paper, the precipitate being washed three or four times with dilute cinchonine solution (30 c.c. of the concentrated solution, 30 c.c. of concentrated hydrochloric acid, 1000 c.c. of water), and finally once only with cold water; in the filtrate the tin may be estimated by the Pearce-Low method. The filter and its contents are heated gently in the original precipitating beaker with 8–10 c.c. of ammonia solution and 25 c.c. of water, stirred to break lumps, filtered into a small beaker, and washed with hot water containing a little ammonia. The excess of the latter is expelled by heating and the liquid treated immediately with 2–3 drops of hydrochloric acid and 8–10 c.c. of the concentrated cinchonine solution and well stirred for some minutes. After 1–2 hrs. the liquid is filtered through an ashless filter containing a little pulped filter-paper and the precipitate washed as before with the dilute cinchonine solution and once with water, dried, ignited in a platinum crucible, and weighed as  $\text{WO}_3$ . If a very exact result is required the precipitate is treated with 1–2 drops of dilute sulphuric acid and a little hydrofluoric acid, the latter being then driven off, 1–2 drops of nitric acid added, and this and the sulphuric acid then expelled by heating; in this way any silica present is eliminated. Cinchonine may be recovered from liquors containing it by rendering them alkaline with ammonia.—T. H. P.

See also pages (A) 256, *Burner gases and oleum* (Sander). 257, *Phosphoric acid* (Stearn and others); *Potassium in silicates* (Morgan); *Polysulphide-sulphur* (Wöber); *Chromic oxide precipitates* (Topresen). 263, *Gold* (Smit). 264, *Chromium in chromite* (Little and Costa). 267, *Annatto in fats* (Brinsmaid). 268, *Coconut oil in soap* (Jungkunz). 270, *Tannin analysis* (Wiley and Krug). 271, *Sucrose* (Browne). 272, *Maltose and lactose* (Le-grand); *Starch* (Lührig); *Diastatic power of malt* (Baker and Hulton). 274, *Alcohol* (Lachman); *Methyl alcohol in sulphite spirit* (Sieber); *Cryoscopy of milk* (Hortvet). 275, *Methyl anthranilate* (Power). 276, *Capsella bursa* (Boruttau and Cappenberg). 279, *Borneol* (Martin). 280, *Mixed and waste acids* (Toussaint).



### Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

#### I.—GENERAL; PLANT; MACHINERY.

##### APPLICATIONS.

- Bain and Keith. Heating and drying apparatus. 9761. Apr. 2.  
 Bartmann. Grinding-mills, disintegrators, etc. 9367. Mar. 29.  
 Bomsel. Preventing incrustation or boiler scale. 10,086. Apr. 6.  
 Cannon. Furnaces or ovens. 9356. Mar. 29.  
 Cook. Chemical and physical synthesis. 9887. Apr. 4.  
 Dine and Sieff. Preparation for removing and preventing scale in boilers etc. 9677. Apr. 1.  
 Hartshorn. Grinding and mixing machines. 9345. Mar. 29.  
 Knoblauch. Tunnel kilns. 10,069. Apr. 5.  
 Laing and Nielsen. Treatment of gases or vapours etc. 9653. Mar. 31.  
 Laurick. Kneaders, mixers, etc. 10,453. Apr. 9.  
 McIntyre. Refining, mixing, etc. machines. 10,095. Apr. 6.  
 Maus. Vacuum filters. 9931. Apr. 4.  
 Mohs. Grinding-mills. 9710. Apr. 1.  
 Vallez. Filter-presses. 10,177. Apr. 6. (U.S., 6,315.)  
 Withers (Etabl. C. H. Candlot Soc. Anon.). Grinding etc. mills. 9392. Mar. 29.

##### COMPLETE SPECIFICATIONS ACCEPTED.

- 29,651 (1919). Harris, and Whiteley and Sons. Drying machines of the travelling-apron type. (160,885.) Apr. 13.  
 32,121 (1919). Brinjes and Goodwin, Ltd., and Seaman. Roller grinding mills. (160,553.) Apr. 6.  
 1607 (1920). Corning Glass Works. Heat treatment of articles. (140,374.) Apr. 13.  
 12,618 (1920). Norske Akt. f. Elektrokemisk Industri. Manufacture of porous material. (143,500.) Apr. 13.  
 16,172 (1920). Metallbank u. Metallurgische Ges. Solidifying in fine powder liquids, pulpy masses, etc. (145,048.) Apr. 13.  
 18,967 (1920). Marks (Ore Roasting Development Co.). Treating solids capable of reaction with a gaseous reagent. (161,103.) Apr. 13.  
 19,290 (1920). Schmidt. Reaction towers. (147,213.) Apr. 13.  
 22,888 (1920). Barbet et Fils et Cis. Recovery of vapours of volatile liquids from air. (149,691.) Apr. 13.  
 24,174 (1920). Eberts. Drying apparatus. (160,717.) Apr. 6.

#### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

##### APPLICATIONS.

- Baty. Process of gas purification. 9868. Apr. 4.  
 Baty, Cox, and Kerr. Gas purification. 10,230. Apr. 7.  
 Biddison. Combustible gas, and generation and burning of same. 10,132. Apr. 6. (U.S., 29,420.)  
 Black, Kime, and Lawson. Refining hydrocarbon oils, spirits, etc. 9436. Mar. 30.  
 Blumner. 9361. See III.  
 Bonnard. Carbonising furnaces or retorts. 9646. Mar. 31.  
 Bowen. Production of artificial fuel. 10,409. Apr. 8.

Brownlee. Synthetically-produced hydrocarbon oils. 9794. Apr. 2. (U.S., 13,513.)

Frentrup, Meyer, and Schubert. Manufacture of consistent fat, cart-grease, etc. from brown coal, peat, slate, paraffin, etc. 9487. Mar. 30.

Frentrup, Meyer, and Schubert. Obtaining components from brown coal, slate, etc. 9488. Mar. 30.  
 Igran Electric Co. 10,302-3. See XXIII.

Macintosh. Destructive distillation of coal. 10,400. Apr. 8. (Australia, 8,420.)

Moncrieff. Retorts. 10,227-8, 10,334. Apr. 7 and 8.

Plauson's, Ltd. (Plauson). Manufacture of pastes or salves from mineral etc. oils. 10,084. Apr. 6.

Verity. Gas-producers. 10,410. Apr. 8.

Wade (Kliva Ges.). 9511. See XII.

Wilson. 9220. See III.

##### COMPLETE SPECIFICATIONS ACCEPTED.

32,236 (1919). Teague. Production of pure carbon. (160,561.) Apr. 6.

32,309 (1919). McComb. Converting liquid hydrocarbons into hydrocarbons of lower boiling point. (160,907.) Apr. 13.

2826, 2828, 2947 (1920). Ellis (Foundation Oven Corp.). Coke ovens. (160,625-6, 160,628.) Apr. 6.  
 7432 (1920). Hurez. Coke ovens. (153,272.) Apr. 13.

30,525 (1920). Klarding. Purifying blast-furnace and generator gases. (153,263.) Apr. 13.

#### III.—TAR AND TAR PRODUCTS.

##### APPLICATIONS.

- Blumner. Continual distillation of tars or oils. 9361. Mar. 29.  
 Wilson. Distillation of tars and oils. 9220. Mar. 29.

##### COMPLETE SPECIFICATIONS ACCEPTED.

15,651 (1920). Pestalozzi. Producing tar of aliphatic compounds or low-temperature tar. (145,408.) Apr. 6.

16,298 (1920). Wohl. Manufacture of phthalic anhydride. (145,071.) Apr. 6.

#### IV.—COLOURING MATTERS AND DYES.

##### COMPLETE SPECIFICATIONS ACCEPTED.

17,848 (1919). Morgan, and British Dyestuffs Corp. Manufacture of hydroxyazo dyes. (160,848.) Apr. 13.

19,392 (1919). Morgan, and Imperial Trust. Manufacture of arylsulphonyl and arylenedisulphonyl derivatives of 1,4-naphthylenediamine and its sulphonic acid. (160,853.) Apr. 13.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

##### APPLICATIONS.

Cassano. Production of cork board. 10,125. Apr. 6. (Ger., 30,420.)

Dreyfus. Manufacture of artificial threads. 10,036. Apr. 5.

Höchstädt. Metallised paper. 9323. Mar. 29.

Masterman. Manufacture of millboard etc. 10,187. Apr. 6.

Roper. Decottonising rags, carbonising cotton, etc. 10,343. Apr. 8.

##### COMPLETE SPECIFICATIONS ACCEPTED.

21,643 (1919). Bouffe. Production of artificial silk threads. (160,859.) Apr. 13.

25,502 (1919). Claessen. Treatment of wood or other substances containing cellulose to obtain cellulose and artificial resin, asphalt, lac, etc. (160,482.) Apr. 6.

25,788 (1919). Hawkins. Machines for washing wool etc. (160,485.) Apr. 6.

1930 (1920). Wells and Marx. Testing paper, board, textiles, etc. (160,619.) Apr. 6.



2999 (1920). Gahler. Producing fibre suitable for spinning from papyrus. (144,249.) Apr. 13.  
 18,945 (1920). Zellkoll Ges. Sizing paper pulp. (147,002.) Apr. 6.  
 18,949 (1920). Merckens. Sizing paper, paste-board, etc. (147,006.) Apr. 13.  
 28,984 (1920). Glanzfaden A.-G. Producing spun material resembling wool or cotton from viscose solutions. (152,351.) Apr. 13.

#### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

##### APPLICATION.

Brandwood and Brandwood. Bleaching, dyeing, etc. textile fibres. 10,331. Apr. 8.

##### COMPLETE SPECIFICATION ACCEPTED.

2890 (1920). Norweb. Fireproofing, metallising, and waterproofing lace and other fabrics. (160,627.) Apr. 6.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

##### APPLICATIONS.

Dossett. Crystallising copper sulphate. 9617. Mar. 31.  
 Ebbw Vale Steel, Iron, and Coal Co., and Thickins. Neutralising and drying sulphate of ammonia. 10,482. Apr. 9.  
 Marks (Pfrschinger Mineralwerke). Increasing bleaching power of silicates. 10,157. Apr. 6.  
 Passmore. Manufacture of magnesium salts. 10,493. Apr. 9.  
 Pike. Treatment of magnesite. 10,205. Apr. 6.  
 Rhenania Ver. Chem. Fabr. Manufacture of sulphurous acid. 10,489. Apr. 9. (Ger., 14.4.20.)  
 Sauvagé. Treatment of artificial gems. 9886. Apr. 4.  
 Soc. l'Air Liquide. Synthesis of ammonia. 9382 and 10,189. Mar. 29 and Apr. 6. (Fr., 30.3 and 7.4.20.)  
 Vernon. 9862. See XI.

##### COMPLETE SPECIFICATIONS ACCEPTED.

16,597 (1915). Buchner. Manufacture of metal hydroxides. Apr. 13.  
 20,021 (1919). Giertsen. See XX.  
 32,539 (1919). Thibault. Converting metallic lead into its oxides. (137,288.) Apr. 13.  
 4694 (1920). Diehl. See X.  
 4976 (1920). Gerber. Manufacture of aluminium nitride. (139,195.) Apr. 6.  
 13,548 (1920). Schweiz. Sodafabrik. Production of caustic soda or soda lye. (144,266.) Apr. 13.

#### VIII.—GLASS; CERAMICS.

##### APPLICATIONS.

Allgem. Elektrizitäts Ges. Firing porcelain etc. in tunnel ovens. 9405. Mar. 29. (Ger., 28.3.20.)  
 Bruner and Clark. Automatic electric heating device for glass furnaces. 9318. Mar. 29. (U.S., 29.3.20.)  
 Bruner and Clark. Electro-fining glass furnace. 10,168. Apr. 6. (U.S., 6.4.20.)  
 Mathys (Richeroux, Lambotte et Cie.). Manufacture of raw plate glass. 9749. Apr. 1.

##### COMPLETE SPECIFICATIONS ACCEPTED.

28,837 (1919). Kelly and Jones. Manufacture of glass and vitreous materials. (160,495.) Apr. 6.  
 10,579 (1920). Broughan (Virginia Plate Glass Corp.). Manufacture of glass. (141,737.) Apr. 13.  
 12,145 (1920). Dimitri and Delaunay. Manufacture of refractory and insulating products. (142,513.) Apr. 13.  
 21,018 (1920). Amsler. Annealing furnace or leer. (160,710.) Apr. 6.

#### IX.—BUILDING MATERIALS.

##### APPLICATIONS.

McKenzie. Manufacture of concrete blocks etc. 10,373. Apr. 8.  
 Mejer. Impregnating composition for cure of efflorescence and sea-salt in brick, mortar, and plaster walls. 9549. Mar. 31.  
 Schneider. Manufacture of coating on stone. 9482. Mar. 30.  
 Smith. Process of curing cement. 9564. Mar. 31.

##### COMPLETE SPECIFICATIONS ACCEPTED.

8004 (1920). Monnoyer and Kirkpatrick. Manufacture of compositions for covering roads etc. (161,061.) Apr. 13.  
 17,944 (1920). Marcussen. Oil-proof concrete bodies and method of making same. (145,673.) Apr. 6.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

##### APPLICATIONS.

Brooke, Cooper, and Kerr. Alloy or solder. 9567. Mar. 31.  
 Coles. Automatic continuous process for coating metal plates or sheets with other metals. 9291. Mar. 29.  
 Coles. Removal of scale or oxide from metallic surfaces. 9292. Mar. 29.  
 Galtarossa and Ongaro. Production of pig iron. 9525. Mar. 30. (Ital., 30.3.20.)  
 Isabellen-Hütte Ges. Treatment of copper alloys. 10,494. Apr. 9. (Ger., 10.4.20.)  
 Kemp, and Wellman Smith Owen Engineering Corp. Metallurgical etc. furnaces. 9734. Apr. 1.  
 Morgan. Substitute for palm oil for tinplate manufacture. 9375. Mar. 29.

##### COMPLETE SPECIFICATIONS ACCEPTED.

21,812 (1919). Lloyd. Roasting and sintering ores etc. (160,477.) Apr. 6.  
 32,363 (1919). Strasser. Deoxidising and refining aluminium. (141,324.) Apr. 6.  
 32,364 (1919). Strasser. Deoxidising and refining copper. (142,441.) Apr. 6.  
 32,539 (1919). Thibault. See VII.  
 4694 (1920). Diehl. Obtaining and utilising the sulphur dioxide from blast-furnace slag. (139,172.) Apr. 13.  
 17,392 (1920). Krupp A.-G. Production of low-carbon ferrochromium. (145,710.) Apr. 13.  
 19,013-4 (1920). Milliken. Alloys. (160,708 and 161,104.) Apr. 6 and 13.  
 30,525 (1920). Klarding. See II.

#### XI.—ELECTRO-CHEMISTRY.

##### APPLICATIONS.

Akt.-Ges. Brown, Boveri, et Cie. Electrically-heated muffle furnaces. 9909. Apr. 4.  
 Niblett. Electrolytic apparatus. 10332. Apr. 8.  
 Vernon. Continuous flow apparatus for making solutions of pure alkali etc. by electrolysis. 9862. Apr. 4.

##### COMPLETE SPECIFICATIONS ACCEPTED.

31,899 (1919). British Thomson-Houston Co., Hastings, and Laycock. Electric furnaces. (160,529.) Apr. 6.  
 467 (1920). Green. Electric accumulators. (160,595.) Apr. 6.  
 12,145 (1920). Dimitri and Delaunay. See VIII.

#### XII.—FATS; OILS; WAXES.

##### APPLICATIONS.

Frentrup and others. 9487. See II.  
 Grün and others. 9652. See XIX.  
 Hull and Steer. Soap. 9333. Mar. 29.

Kestner Evaporator and Engineering Co., and Reavell. Extraction of oils, fats, waxes, etc. 9704. Apr. 1.  
Marks (Pferschinger Mineralwerke). 10,167. See VII.  
Mizusawa and Yamamoto. 9753. See XIX.  
Morgan. 9375. See X.  
Wade (Kliva Ges.). Manufacture of fatty acids from hydrocarbons. 9511. Mar. 30.  
Webster. Treating gaseous constituents of fatty oils, gums, resins, etc. 9434. Mar. 30.

## COMPLETE SPECIFICATIONS ACCEPTED.

29,001 (1919). Efront. See XVIII.  
29,973 (1919). Chadbourne. Manufacture of saponaceous compositions. (160,892.) Apr. 13.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

## APPLICATIONS.

Cooper. Washable waterproof water paint. 9543. Mar. 31.  
Debn (Satow). Manufacture of insoluble condensation product. 10,214. Apr. 6.  
Parker. Metallic water paint. 9306. Mar. 29.  
Webster. 9434. See XII.

## COMPLETE SPECIFICATION ACCEPTED.

25,502 (1919). Claessen. See V.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

## COMPLETE SPECIFICATION ACCEPTED.

29,172 (1919). Peachey and Skipsey. Obtaining vulcanised rubber goods and materials containing substances adversely affected by certain vulcanising processes. (160,499.) Apr. 6.

## XV.—LEATHER; BONE; HORN; GLUE.

## APPLICATION.

Plausons, Ltd. (Plauson). Manufacture of glue. 9762. Apr. 2.

## COMPLETE SPECIFICATIONS ACCEPTED.

14,626 (1920). Elektro-Osmose A.-G. Process for tanning or impregnating materials. (143,921.) Apr. 13.  
32,891 (1920). Lambeek. Making the colouring of natural horn water-resisting. (154,200.) Apr. 13.

## XVI.—SOILS; FERTILISERS.

## APPLICATIONS.

Lo Monaco. Chemical fertilisers. 10,315. Apr. 7.  
Soc. l'Azote Français. Manufacture of fertilisers. 9372. Mar. 29. (Switz., 29.3.20.)

## COMPLETE SPECIFICATION ACCEPTED.

12,495 (1919). Morel. Manufacture of assimilable phosphates. (160,847.) Apr. 13.

## XVIII.—FERMENTATION INDUSTRIES.

## COMPLETE SPECIFICATIONS ACCEPTED.

29,001 (1919). Efront. Manufacture of pressed yeast from distillers' grains, oil cake, etc. (160,496.) Apr. 6.  
32,245 (1919). Townsend. Manufacture of fermented liquors. (160,562.) Apr. 6.

## XIX.—FOODS; WATER PURIFICATION; SANITATION.

## APPLICATIONS.

Adam and Gair. Removing oil from condensation waters. 10,310. Apr. 7. (Fr., 15.4.20.)  
Dienst. Sterilising flour and grits from cereals. 9806. Apr. 2.

Fyers and Looker. Preservative treatment and refrigeration of fish. 9827. Apr. 2.  
Green. Centrifugal sewage purification. 9776. Apr. 2.

Grün, and Schicht A.-G. Manufacture of butter fat and lard. 9652. Mar. 31. (Czecho-Slov., 1.4.20.)  
Imperial Trnst, and Piqué. Cooling and freezing fish etc. 9485. Mar. 30.

Mizusawa and Yamamoto. Preparing odourless and colourless oil and flour from beans. 9753. Apr. 1.

## COMPLETE SPECIFICATIONS ACCEPTED.

7272 (1915). Wehner. Apparatus for separating gases, iron, manganese, etc. from water. Apr. 6.  
31,354 (1919). Hawker. Insecticides, fungicides, animal dips, etc. (160,511.) Apr. 6.  
570 (1920). McDougall and Howles. Sheep-dips and cattle-washes. (160,597.) Apr. 6.  
21,856 (1920). Drayer and Hanson. Preserving eggs. (161,110.) Apr. 13.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

## APPLICATIONS.

Farbw. vorm. Meister, Lucius, u. Brüning. Manufacture of dialkylaminoethyl- $\beta$ -aracyl oxybutyric acid esters. 10,508. Apr. 9. (Ger., 10.4.20.)  
Haddan (Jenny). Converting organic acids into esters. 9927-8. Apr. 4.  
Plauson. 10,084. See II.

## COMPLETE SPECIFICATIONS ACCEPTED.

20,021 (1919). Giertsen. Production of urea or ammonia from cyanamide. (160,857.) Apr. 13.  
32,368 (1919). Darrasso Frères et Cie. Manufacture of valeric acid and alkali valerates. (137,064.) Apr. 13.  
16,298 (1920). Wohl. See III.  
17,780, 33,206-7 (1920). Kolsborn. Manufacture of derivatives of *p*-aminophenol and of its *o*-alkyl ethers. (145,614, 155,575-6.) Apr. 6.  
29,956 (1920). Wacker Ges. f. Elektrochem. Ind. Manufacture of dichlorethylene. (156,080.) Apr. 13.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## APPLICATIONS.

Martinez. Colour photography and/or cinematography. 9993. Apr. 5.  
Ott. Solution for coating kinematograph films etc. 10,202. Apr. 6.

## COMPLETE SPECIFICATION ACCEPTED.

31,997 (1919) and 10,576 (1920). Greene, Thomson, and Colour Photography, Ltd. Colour photography. (160,540.) Apr. 6.

## XXIII.—ANALYSIS.

## APPLICATIONS.

Helps. Calorimeters. 10,323. Apr. 7.  
Igranic Electric Co. (Cutler-Hammer Manuf. Co.). Measuring calorific value of combustible gases etc. 10,302-3. Apr. 7.  
Krogh and Pedersen. Recording analysing apparatus for automatic analysis of gases. 9560. Mar. 30.

## COMPLETE SPECIFICATIONS ACCEPTED.

19,518 (1919). Rodhe, and Svenska Akt. Mono. Gas-analysing apparatus. (160,854.) Apr. 13.  
32,597 (1919). Davis and Rosling. Apparatus for analysing gases. (160,930.) Apr. 13.  
2680 (1920). Svenska Akt. Mono. Gas-analysing apparatus. (138,355.) Apr. 13.

## I.—GENERAL; PLANT; MACHINERY.

*Evaporation, concentration, and desiccation of all organic or mineral substances; Process of —.* A. Sartory, L. Scheffler, P. Pellissier, and C. Vaucher. *Comptes rend.*, 1921, 172, 744–746.

THE apparatus, which is sketched, is so arranged that the substance is submitted to a current of air deprived of moisture at a temperature near to 0° C., to avoid risk of change or decomposition of the substance being dried or of the liquid being evaporated or concentrated. The substance to be dried is placed in a box made of lattice-work, mounted on a shaft and rotated in the current of air. The air is first cooled below 0° C. to remove its moisture, and then warmed up to the temperature suitable for the substance under treatment. Thus to remove the water (80% by weight) from 15 kg. of fresh mest in 10 hrs. at 5° C., the air was first cooled to -8° C. to remove its moisture and then warmed to 5° C. This necessitated the passage of 1300 cub. m. of air per hr., the production of 9000 frigories per hr. for cooling the air to -8° C., and of 5000 cal. per hr. for subsequently warming it to 5° C.—W. G.

*Refrigeration machines; Causes and prevention of the formation of non-condensable gases in ammonia absorption —.* E. C. McKelvy and A. Isaacs. U.S. Bureau of Standards, Techn. Paper No. 180, 25.10.20. 10 pages.

NON-CONDENSABLE gases in ammonia absorption refrigerating machines consist of nitrogen, hydrogen, and to a minor extent of oxygen. The presence of nitrogen is due to leaks of air into the system, and hydrogen when present is due to corrosive action of the liquid ammonia on the metal of the plant. Oxygen leaking into the plant is quickly used up. Impurities such as carbonates, acetates, and acetonitrile increase the corrosive action of ammonia liquor at ordinary operating temperatures to such an extent as to account readily for the hydrogen found in many plants. Where the quantity of carbonate present does not exceed 0.003%, the corrosive action of the liquor is not marked. For the determination of small quantities of carbonate in the ammonia solution, 100 g. of the latter is added to excess of sulphuric acid, the liberated carbon dioxide is absorbed in barium hydroxide solution, the precipitated barium carbonate washed and dissolved in hydrochloric acid, and the barium precipitated as sulphate. Gas formation due to corrosive action of the ammonia is prevented by adding 0.2% by weight of sodium or potassium bichromate to the generator charge.—J. S. G. T.

## PATENTS.

*Filter elements; Method of making — [for separating bacteria etc.].* E. de Haën, Chem. Fabr. "List" G.m.b.H. E.P. 134,228, 23.10.19. Conv., 4.12.17.

A CELLULOSE ester is dissolved in a double solvent, volatile at ordinary temperature (e.g., a mixture of acetone, chloroform, or ethyl acetate with glacial acetic acid, an alcohol, or an ether), and the solution, in the form of a thin layer, allowed to evaporate in a moist atmosphere, with or without previous addition of water to the solution. The air for evaporation may be circulated through an apparatus which removes the solvent and re-conditions the air as regards moisture content, and the film, which will be crinkly, may be flattened by stretching in hot water or steam.—B. M. V.

*Pressure filters; Flushing apparatus for use with —. Filter-frame for use with pressure filters.* L. J. Martel, Assr. to Martel Filter Co., Inc. U.S.P. (A) 1,370,469 and (B) 1,370,470, 1.3.21. Appl., 11.3.20.

(A) An oscillating spray pipe provided with a number of nozzles projects into the body of the filter to enable the filter cloths to be sprayed. The pipe is enclosed in a tubular sheath which normally covers the nozzles, but is slotted so as to expose the nozzles when the pipe is turned. (B) A filter-frame comprises a sheet-metal frame enclosing filter sheets of woven wire, the upper edge of the frame being formed as a strap through which a pipe for liquid supply passes, and supports the frame. The pipe is slotted longitudinally, and one of the wire sheets projects into the slot.—W. F. F.

*Metallic filter gauze; Process for manufacturing —.* J. Rahtjen. G.P. 330,715, 15.1.19. Addn. to 329,061 (J., 1921, 203 A).

METAL gauze mixed with material such as cotton wool, wool, silk, linen, or nettle, ramie or similar fibre, is worked up into the form of filter cloth and is coated with a uniformly porous layer of metal by means of a metal-spraying device. The gauze is suitable for filtering oily and alcoholic liquors containing very small particles.—J. S. G. T.

*Gas purifying apparatus.* C. Bourdon. E.P. 139,758, 11.2.20. Conv., 6.3.19.

SOLID matter is removed from the gas by centrifuging, and is discharged into a torus-like passage formed by parts of the peripheral walls of the fan casing. A tubulure connects the inlet to the fan with a conduit conveying the gas to be purified, the conduit being arranged in the interior of a chimney shaft through which the purified gas is discharged into the atmosphere or delivered to apparatus where it is to be used. The gas can be short-circuited without purification, if desired, by means of a suitable valve.—A. G.

*Dry cleaning [separating the solids from] gases and vapours, more especially furnace gases; Filter for —.* E. Dänhardt. E.P. 148,800, 10.7.20. Conv., 25.2.18.

FILTERS, which may be used at elevated temperatures, are constructed of metallic wires or perforated plates interwoven with mineral fibres, the surface of the latter being roughened, on the side upon which the gas impinges, by devices such as are used in the textile industry.—B. M. V.

*Gases; Apparatus for separating dust and the like from —.* R. Boby, Ltd., and M. Jennings. E.P. 160,100, 27.5.20.

DUST-LADEN air or gas is caused to pass down and up several times inside a vertical container, the changes of direction being effected by domes or hoods provided with conical skirts, so that the passage for the gas gradually increases in cross-sectional area. The inlet pipe for the gas is at the centre and the outlet at the circumference of the container, and the separated dust is collected and drawn off at intervals. If desired, the gas may be divided into two or more streams by means of a short pipe or pipes inserted upwards into the bottom of the first downtake (inlet) pipe, the streams being kept apart by additional hoods and skirts till just before reaching the outlet.—B. M. V.

*Gases or vapours; Process for treatment of — with liquids.* H. Roser. G.P. 330,071, 17.4.14.

INTIMATE mixing, efficient distribution and transportation of the gas or vapour and the liquid are

effected by introducing them into a vessel, formed somewhat similar to a hydraulic compressor, and connected with a fall tube.

*Electrical separation of dust from gases and vapours; Apparatus for* —. A.-G. Brown, Boveri & Co. G.P. 331,143, 24.9.19.

IN an apparatus consisting of a dust chamber in which electrodes of opposite polarity are arranged in rows, each electrode extends within a guiding framework to a width greater than the chamber, and can be displaced to either side for cleaning so that the part of the electrode within the chamber is changed from time to time.—C. I.

*Grinding or crushing mills.* H. Cramm. E.P. 145,599, 29.6.20. Conv., 15.5.14.

A ROTATING cylindrical drum is flattened in three or more places to give it a roughly polygonal section (with rounded angles), and is provided with a loose grinding roller of nearly the same length as the drum.—B. M. V.

*Ball-mill.* G. B. Allison, Assr. to G. H. Beesmyer. U.S.P. 1,370,259, 1.3.21. Appl., 4.2.20.

A HORIZONTAL rotating drum contains an inner concentric drum attached at one end to the end wall of the outer drum, and having a separate end wall at the other end spaced from the outer drum. The annular space between the drums is provided with a number of ball races side by side, with freely movable grinding balls. Ports are provided between the inner and outer drums, and hollow trunnions communicate respectively with the inner drum and the annular space.—W. F. F.

*Boiling pans and mixing machines.* J. MacLachlan, G. Stewart, and A. E. Clark. E.P. 159,924, 7.11.19.

ONE set of stirring and scraping gear may serve two or more boiling pans by supporting the former upon a radial arm which can be swung round a vertical column.—B. M. V.

*Ring furnace and drying plant.* A. E. H. Beyer. E.P. 160,328, 16.1.20.

IN a combined ring furnace and drying plant the conduits between the separate divisions of the furnace and the individual chambers of the drying apparatus and the chimney are so arranged that air may be caused to flow from any one division of the furnace through any one chamber, or *vice versa*, and thence to the chimney; or from any one division of the furnace or chamber to the chimney, either direct or through another division or chamber.—H. H.

*Furnace.* J. U. McDonald. U.S.P. 1,370,139, 1.3.21. Appl., 7.9.20.

A CRUCIBLE furnace is provided with a bottom sloping towards the front, and with an arched top having openings to admit crucibles. A fuel-supply pipe extends through the front wall, and the rear wall has a ridge extending forward to cause the combustion products to divide and pass along the two side walls.—W. F. F.

*Furnace or oven.* H. B. Cannon. U.S.P. (A) 1,371,773 and (B) 1,371,774, 15.3.21. Appl., 29.3.20.

(A) AN oven has a space for subjecting articles to radiant heat and a combustion chamber in operative relation to such space, but not communicating therewith, the combustion chamber being formed principally of carborundum. (B) A combustion chamber is provided, intermediate of its height, with a partition extending from the front towards and spaced from the rear, the top of the chamber being provided with an outlet near the front. The chamber is formed principally of carborundum, and

means are provided for supplying a combustible mixture into the front of the chamber beneath the partition.—H. H.

*Dissolving, crystallising, and calcining apparatus; Combined* —. A. Roy, Assr. to J. L. Sharkey. U.S.P. 1,369,840, 1.3.21. Appl., 12.12.18.

DISSOLVING, crystallising, drying and calcining chambers are arranged in series. A heating medium is supplied to the calcining chamber, and the excess of heat is utilised successively in the drying, crystallising, and dissolving chambers.—W. F. F.

*Continuous solution, washing or lixivation of salts; Apparatus for the* —. H. Pappée. G.P. 327,687, 20.9.18.

A FILTER bed composed of fine-grained material, such as sand, is disposed immediately beneath the vessel containing the solution to be treated. The surface of the filter bed is funnel-shaped, the apex being directed downwards, and a number of spaced overlapping plates are laid thereon. A number of sliding bars extending from top to bottom of the funnel-shaped surface form a grid with the plates. An archimedean screw arranged within a tube extending upwards from the apex of the funnel serves to remove any residue from the filter bed. The device can be operated with the formation of very little sludge.—J. S. G. T.

*Centrifugal clarifier.* M. Leitch, Assr. to The De Laval Separator Co. U.S.P. 1,370,326, 1.3.21. Appl., 19.8.19.

LIQUID is delivered into the top of a rotating vertical vessel having a contracted central opening at the bottom, from which it passes to an outer containing vessel which also rotates and in which the liquid is subjected to a greater centrifugal force.—W. F. F.

*Desiccating-machine.* G. C. Contant, Assr. to J. Decker, jun. U.S.P. 1,371,071, 8.3.21. Appl., 18.7.19.

A TRAVELLING metallic belt is arranged within a partly exhausted container, the material to be desiccated being directed on to the upper run of the belt, and heating devices being disposed below and almost in contact with both upper and lower runs.—H. H.

*Evaporator.* T. M. Pusey. U.S.P. 1,371,115, 8.3.21. Appl., 18.9.19.

AN evaporating vessel is supported in a structure which forms a receptacle at the bottom thereof for a fluid heating medium. External to the vessel, but within the structure, are circulating means, connected with the bottom of the vessel for the downflow of the fluid contents of the vessel, and also connected with the vessel above the bottom for the return of the fluid thereto.—H. H.

*Evaporator.* J. C. Grêre. U.S.P. 1,371,784, 15.3.21. Appl., 24.8.20.

TWO evaporating vessels are connected by a conduit in which is a chamber from which steam is returned to the first vessel. A second chamber in the conduit is in communication at its top and bottom with the top and bottom, respectively, of the second vessel, and this chamber contains a continuous spiral passage with perforated transverse partitions. A valve between the two chambers is controlled by the height of the fluid in the first chamber.—W. J. W.

*Refrigerating system, and method of operating the same.* G. A. Gase and E. A. Seymer. U.S.P. 1,371,235, 15.3.21. Appl., 8.1.17.

IN a method of refrigeration in which a refrigerating medium is liquefied with evolution of heat at

one portion of a closed system and vaporised with absorption of heat at another portion, a pressure is maintained in the system by an inert gas in order to control the absorption of heat.—H. H.

*Solvent-recovery apparatus.* W. K. Lewis and W. Green. U.S.P. 1,371,914, 15.3.21. Appl., 8.5.19.

AN apparatus for removing and recovering a volatile inflammable solvent from a material containing it comprises a drying chamber, means for supporting the material in the chamber, a burner, means for conducting flue gas from the burner into the chamber to take up the vapour of the solvent, means for cleaning the flue gas on its way to the chamber, and means for recovering the solvent from the gas leaving the chamber.—H. H.

*Volatile solvents; Removal of — from material treated therewith.* H. Bollmann. U.S.P. 1,371,546, 15.3.21. Appl., 9.12.19.

SEE E.P. 154,044 of 1919; J., 1921, 33 A.

*Condensers and boilers; Disincrustant for cleaning —.* J. Urruty, Assr. to Urruty et Cie. U.S.P. 1,371,584, 15.3.21. Appl., 11.12.18.

SEE E.P. 121,590 of 1918; J., 1919, 704 A.

*Furnace; Rotary —.* F. Derneden. U.S.P. 1,372,406, 22.3.21. Appl., 16.9.19.

SEE E.P. 156,835 of 1919; J., 1921, 169 A.

*Furnaces; Tilting —.* A. Imbery. E.P. 160,247, 15.12.19.

*Density of liquids; Apparatus for indicating the — and automatically grading the same according to their density.* J. L. Mennell, and C. Butters and Co., Ltd. E.P. 160,293, 24.12.19.

*Grinding mills; [Mounting and adjusting rollers in] roller —.* Brinjes and Goodwin, Ltd., and C. J. Seaman. E.P. 160,553, 22.12.19.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal; Determination of heat of carbonisation of — in the calorimeter.* H. Strache and H. Grau. Brennstoff-Chem., 1921, 2, 97—99.

OTTO (Diss., Breslau, 1914) determined the heat of coking by using an electric resistance furnace embedded in asbestos in an iron vessel, the furnace being kept at a constant temperature and a steady temperature gradient being maintained by immersing the iron vessel in a cold water bath at constant temperature. The fall in temperature of the oven after introduction of the coal charge is compensated for by extra current input, and the energy required for this purpose is measured, and from it the heat of coking is calculated. By this method the gross heat of carbonisation (referred to water and tar at 0° C.) of a certain coal was found to be 260 cal. per g. Grau has determined this value directly by carbonising 1 g. of coal in a copper cylinder, fitting into a quartz tube furnace, wound with constantan, the whole being contained in a Berthelot-Mahler bomb which was filled with nitrogen at the commencement of the experiment. The temperature was measured by means of an iron-constantan couple. A temperature of 700° C. was attained in 10 mins. and current and voltage at the furnace terminals were measured every 15–20 seconds. Values are quoted for coals, brown coals, lignites, wood, and cellulose ranging from 8 cal. per g. (absorbed) for coal with 26.27% of volatile matter to 265 cal. per g. (evolved) for beech wood with 76.94% of volatile matter. The

gross heat of carbonisation seems to be proportional to the oxygen content of the coal.—A. G.

*Coal and other solid fuels; Oxidation of — under pressure.* F. Fischer and H. Schrader. Ges. Abhandl. Kennt. Kohle, 1919, 4, 342—359. Chem. Zentr., 1921, 92, II., 422.

THE percentages of fuel converted into oxidation products by a pressure oxidation for 3 hrs. at 200° C. in presence of sodium carbonate solution reached, for older coals 10%, for younger coals above 30%, for lignite and peat above 90%, for wood 75%, and for cellulose almost 100%. Only a moderate proportion was converted into carbon dioxide. Humic acids, clear oily acids, higher and lower fatty acids, and oxalic acid were obtained. The oily acids contained aromatic compounds, for when distilled some phthalic anhydride was obtained. No mellic acid was obtained. Insoluble humic acids of high molecular weight, which form the first stage of oxidation of a portion of coal and wood substance and also are present in lignites and peat, are transformed first into more soluble though still dark-coloured humic acids and then into lighter-coloured acids of progressively smaller molecular weight. Wood such as box and fir yielded humic acids, but pure cellulose none.—H. J. H.

*"Oilcoals" [deposits in internal combustion engines etc.]; Composition and formation of —.* J. Marcusson. Brennstoff-Chem., 1921, 2, 103—104.

THE author gives the name "oilcoal" to the carbonaceous deposits formed in internal combustion engines, compressors, steam engines, etc. Such deposits generally consist of an oily portion soluble in benzene, a brittle asphaltic constituent insoluble in benzene, a coaly portion, and mineral ash. The coaly portion, which has previously been considered as insoluble in organic solvents, has been shown by the author to be soluble if a large quantity of solvent is used. Asphaltogenic acids or their anhydrides were separated from the solution and the residue consisted of "carbenes" or "carboids" (products of oxidation of mineral oil). A deposit occurring in a hydrogen compressor consisted of 6% of oil soluble in benzene, 8% of a black solid material (half of which was saponifiable), soluble in chloroform, and 86% of material insoluble in chloroform. The 27% of ash contained tin oxide and sodium carbonate. The theory advanced is that, by previous long use of the oil, resinification and the formation of asphaltenes, carbenes, and asphaltic acids has taken place. These acids have attacked the metal with the formation of tin soaps. At the same time an increase in the amount of alkali soap present in the oil has taken place. This accounts for the presence of alkali carbonate in the ash of the deposit.—A. G.

*Coal gas and coke-oven gas; Determination of benzene hydrocarbons in —.* E. Berl, K. Adress, and W. Müller. Z. angew. Chem., 1921, 34, 125—127.

A LAYER of dry charcoal is placed in a U-tube which is provided with gas inlet and outlet tubes and taps. A current of gas is then passed over the charcoal at a rate which depends on its content of benzene hydrocarbons, but is approximately 250 l. per hr. When absorption is complete, the U-tube is connected at one end with a steam supply and at the other end with a condenser and measuring burette; the tube is heated in a bath to about 110°–120° C., and steam is passed through for  $\frac{1}{2}$  hr., the distilled hydrocarbons being measured in a burette. The method gives more accurate results than the freezing, paraffin oil, and dinitrobenzene methods, and is further applicable,

where the above are nonsuitable, in cases where there is only a small amount of benzene hydrocarbons present.—W. J. W.

*Paraffin wax; Pressure oxidation of* —. F. Fischer. Ges. Abhandl. Kennt. Kohle, 1919, 4, 35–47. Chem. Zentr., 1921, 92, II, 377.

THE oxidation was conducted in a horizontal autoclave provided with an agitator, 200 g. of hard paraffin wax being heated to 170° C., while a current of air at 30 atm. pressure (200 l. per hr.) was passed through. 800 c.c. of 2.5N sodium carbonate solution was present, and oxidation was continued until one half of this was neutralised by the fatty acids formed. The residue was not pure paraffin wax. Fatty acids of average molecular weight 250, insoluble in water and suitable for soap making, were obtained. Acids soluble in water were also produced and a considerable quantity of carbon dioxide. The sodium salts of the higher acids suffer some decomposition during the reaction. Some light paraffin oils were obtained.

—H. J. H.

*Pressure oxidation of paraffin wax; Influence of various factors during the* —. F. Fischer and W. Schneider. Ges. Abhandl. Kennt. Kohle, 1919, 4, 48–93. Chem. Zentr., 1921, 92, II, 377–378.

THE influence of various factors was ascertained by variation of the conditions of the normal experiment described in the preceding abstract. The degree of filling of the autoclave was important. Rise of temperature affected the colour of the products as well as the speed of oxidation. Quantity of air influenced the molecular weight of the acids. Increase of air pressure caused acceleration of the reaction. Undue prolongation of the oxidation caused a secondary decomposition of the acids produced. Speed of stirring, quantity and concentration of alkali had little influence. The use of lime as base was found undesirable. The quantity of paraffin influenced the colour and yield of acids. Iron, manganese, and copper were the best catalysts, but cerium, titanium, fuller's earth, and charcoal gave products with the best colour. The addition of toluene and xylene improved the yield of solid fatty acids, although neither of these solvents, in the absence of paraffins, is oxidised under the experimental conditions. When the apparatus was lined with glass or lead the reaction was retarded and the colour of the product improved. Paraffins obtained from lignite, coal, peat, or mineral oil are all suitable for pressure oxidation, although the first two are more resistant. Paraffin oils yielded liquid acids insoluble in water. In the absence of alkalis paraffin wax was more extensively oxidised than in their presence. The yield of fatty acids was 74% of the paraffin consumed, but their colour was dark brown.—H. J. H.

*Paraffin wax; By-products of the pressure oxidation of* —. W. Schneider. Ges. Abhandl. Kennt. Kohle, 1919, 4, 101–117. Chem. Zentr., 1921, 92, II, 378–379.

In the treatment of the product obtained in oxidation experiments such as described in the preceding abstracts, the cake of unchanged paraffin was separated and the residue worked up to a solid soap. The presence of water-soluble salts of lower fatty acids in the crude product would, if these were not removed, render the soap hygroscopic and impart a disagreeable odour. The solution of salts of lower acids, however, lathers well, and such solutions are suitable for the laundry and scouring purposes. The soda solution of these acids was filtered off from the insoluble salts and evaporated to dryness. The residue contained 1% of neutral material

soluble in benzene, and 54% of fatty acid salts, equivalent to 46% of fatty acids. Half were insoluble in water, ranging from capric to lauric acids, the rest of the acids being soluble. Some oils separated on cooling the compressed air after leaving the autoclave. None boiled below 150°, and 67% between 150° and 250° C. Neutral substances containing oxygen, e.g., alcohols, aldehydes, and ketones, were present. Solid substances, of similar chemical character, also containing oxygen, were found in the cake of residual unoxidised paraffin. The liquid portions had a fruity smell, while the solid portion resembled a true wax rather than paraffin.—H. J. H.

*Paraffin wax; Treatment of the acids obtained in the pressure oxidation of* —. W. Schneider and A. Jantsch. Ges. Abhandl. Kennt. Kohle, 1919, 4, 118–130. Chem. Zentr., 1921, 92, II, 379.

THE oxidation products of paraffin obtained as described in the preceding abstracts were examined with a view to identifying the acids. To effect a separation the ethyl esters were prepared. The acids conformed to the general formula  $C_nH_{2n}O_2$ , but the presence of hydroxy-, keto-, and dibasic acids could not be established. For the acids identified the values of  $n$  were 19, 17, 15, and 13, the odd numbers of carbon atoms being predominant. On the other hand, in fats and oils the acids have mainly an even number of carbon atoms.

—H. J. H.

*Paraffin wax; Oxidation of* — by air at ordinary pressure. W. Schneider. Ges. Abhandl. Kennt. Kohle, 1919, 4, 132–142. Chem. Zentr., 1921, 92, II, 376–377.

EXPERIMENTS on the oxidation of paraffin wax were made in glass and iron vessels. The yield of fatty acids insoluble in water was only 2–3% in glass vessels, but 64% in iron. Mercuric oxide had no catalytic effect. The formation of acid anhydrides is indicated by the presence of products soluble in hot sodium hydroxide solution, but insoluble in sodium carbonate. The reaction velocity is very much smaller than when pressure is employed.

—H. J. H.

*Montan wax; Pressure oxidation of* —. F. Fischer and W. Schneider. Ges. Abhandl. Kennt. Kohle, 1919, 4, 180–209. Chem. Zentr., 1921, 92, II, 376. (Cf. Fischer and Tropsch, J., 1920, 55 A.)

PRESSURE oxidation experiments with air made on crude montan wax, with and without a preliminary saponification, indicated that a degradation to medium and lower fatty acids occurred, although montan wax is distinctly more inert in this respect than paraffin wax. The soaps obtained from the reaction products did not lather so well as those derived from paraffin. The yield was also lower. Fatty acids once formed are rapidly broken down, thus accentuating the poor yield.—H. J. H.

*Montan wax.* Schneider and Jantsch. See III.

*Simmanee calorimeter.* Gray and Blackie. See XXIII.

#### PATENTS.

*Peat fuel; Preparation of* — for producer or other gas for power purposes. S. C. Davidson. EP. 159,996, 11.12.19.

A portion of the peat is taken straight from the bog and is worked up to a putty-like consistency. It is then blended with a proportion of powdered dry peat and slaked lime and pressed into blocks.

—A. G.

*Artificial fuel; Manufacture of* — J. W. Leadbeater. E.P. 160,042, 17.1.20.

BLACK peat, straight from the bog, is finely disintegrated and mixed with ground caustic lime, with finely ground and dried pitch or tar, or both, and with a tar or mineral or fuel oil or fatty oils or a combination of two or more of such oils, to which mixture is added finely ground coal.—A. G.

*Briquettes; Coal or like — and the manufacture thereof.* A. A. Dale. E.P. 160,279, 18.12.19.

COAL is finely ground together with about 10% of lime or other alkaline-earth oxide in water. The mixture is allowed to stand out of contact with atmospheric or other carbon dioxide until hydration is complete and the mass is then briquetted. Ferric oxide or manganese oxide may be added to the extent of 0.5% or 1.0% to promote combustion. In order to render the briquettes weatherproof they may be dipped in sodium silicate solution.

—A. G.

*Pulverised fuel furnaces.* J. E. Kennedy. E.P. 144,724, 11.6.20. Conv., 9.3.18.

THE relatively long combustion chamber of the furnace has parallel longitudinal walls and no internal obstructions. The air blast carrying the pulverised fuel enters the combustion chamber centrally and in a direction parallel to the longitudinal walls, the fuel supply nozzle extending within the combustion chamber. The combustion chamber is completely open to the atmosphere at the air blast inlet end.

—A. G.

*Shaft furnace for fuels rich in water and gas.* R. Bergmans. G.P. 330,183, 13.11.18.

THE upper part of the shaft is divided so as to form one chamber where fresh fuel is carbonised and another where the gas produced is burnt, the latter communicating with the gas off-take main.

—H. J. H.

*Coke or carbon; Separation of — from slag or the like.* Soc. "Le Coke Industrielle." E.P. 156,562, 10.6.20. Conv., 7.1.20. Addn. to 120,932 (J., 1919, 672 A).

THE separating compartments described in the original patent are adjustable at any angle by means of joints and slides, for the purpose of adjusting the sizes of the openings through which the ascending currents of water rise. The inlet pipes for the ascending currents of water comprise concentric tubes, the inner tube being provided with orifices of decreasing size communicating with the outer tube, which is provided with small holes of equal size communicating with the respective separating compartment. The outer plate is a grating contiguous with which is a straight or curved perforated plate.

—A. G.

*Coke oven.* C. E. Lucke, Assr. to L. and A. A. Wilputte. U.S.P. 1,371,315, 15.3.21. Appl., 30.8.17. Renewed 13.8.20.

A REGENERATIVE coke oven has parallel horizontal coking chambers, with heating walls between the chambers, and in each heating wall two groups of heating flues and an upper horizontal passage to which the upper end of each of the flues in both groups is connected. A set of regenerators runs transversely to, and beneath the chambers and heating walls to which the lower ends of the flues in one group of each of the various heating walls are connected, and another set of similarly located regenerators is connected with the lower ends of the remaining flues, each regenerator being connected with a part only of the flues in the group of each heating wall with which the corresponding set of regenerators is connected. Separate

waste gas outlet channels, running parallel to the regenerators, are provided for the various regenerators, and also a common stack flue with which these channels are individually connected at the end of the oven.—A. G.

*Gas manufacture.* E. V. Hayes-Gratze. E.P. 160,202, 10.7.20.

THE material to be carbonised is fed through a number of tubes heated by the exhaust gases of an internal combustion engine.—A. G.

*Purifying gas; Material for —. Material for purifying gas and process for manufacturing the same.* J. E. Hutchinson, Assr. to J. F. Henderson. U.S.P. (a) 1,370,494 and (b) 1,370,495, 1.3.21. Appl., (a) 8.12.19, and (b) 15.2.19. (b) Renewed 27.8.20.

(a) THE purifying material consists of a porous mass, the surface of the pores being coated with the absorbent. (b) The material is made by incorporating hydrated ferric oxide with silica and cement to form a porous concrete.—A. G.

*Heating-value indicator for illuminating gas.* E. J. Brady, Assr. to U. G. I. Contracting Co. U.S.P. 1,370,945, 8.3.21. Appl., 29.7.19.

A HEATING-VALUE indicator for gas comprises a burner provided with a tip and a fuel inlet, and closed to the atmosphere between the inlet and tip. Means are provided for supplying a mixture of air and gas at the fuel inlet in changing ratio, and for ascertaining the air: gas ratio of the mixture such that the luminous tip of the flame at the burner ceases to exist. (Cf. J., 1919, 938 A.)—J. S. G. T.

*Oil from oleiferous sandstone, shale, or the like; Apparatus for the extraction of —.* T. H. Oswald and A. D. Dixon-Brown. E.P. 160,114, 7.7.20.

THE material is charged by means of a screw conveyor into one end of a rotating cylindrical still provided in the interior with radial fins, and passes down the still, which is slightly inclined to the horizontal, to the lower end, whence it is removed by another screw conveyor to an outlet pipe dipping into a water-seal. Superheated steam passes into the still at the lower end through the hollow centre of the screw conveyor, and leaves the apparatus together with the products of distillation through a similar opening at the upper end.—L. A. C.

*Bituminous shale; Process for treating —.* J. H. Ginet. U.S.P. 1,371,160, 8.3.21. Appl., 31.10.19.

THE shale is finely ground and is showered into a heated retort for the entire period necessary to distil all of the volatilisable products.—A. G.

*Hydrocarbon oils; Cracking of —.* R. C. Holmes and F. T. Manley. E.P. 160,200, 8.10.19.

EACH still in a series of vertical cracking stills is heated by furnace gases over the middle portion of its height, and is provided with rotating scrapers to remove carbon deposits from the interior. Valved outlets are attached to the bottom of the stills for withdrawing residual oil, which passes through a heat exchanger to preheat the inflowing oil, and the stills are connected in series at the top by two sets of pipes at different levels to equalise respectively the level of the oil and the vapour pressure in the stills. The oil is pumped into the bottom of the first still, and vapour passes from the last of the series into an air condenser, which returns condensed liquid to the first still, and thence into a water condenser. The pressure within the apparatus is maintained at about 150–200 lb. per sq. in. by a regulator attached to the end of the water condenser.—L. A. C.



*Hydrocarbons; Converting heavy into lighter* — L. W. Gould. From Universal Oil Products Co. E.P. 160,236, 12.12.19.

Oil, e.g., gas oil from Kansas or Oklahoma crude oil, is pumped through horizontal tubes of 4 in. diam., heated to 750°–800° F. (400°–460° C.), and thence into horizontal expansion tubes of 10 in. diam. The residual oil is withdrawn from the latter tubes at such a rate that the tubes are kept about half full, and the vapour passes to an air condenser, which returns condensed oil to the heating tubes, and thence to a water condenser. The oil during the whole of the treatment is maintained under a pressure of about 100 lb. per sq. in. —L. A. C.

*Material [petroleum hydrocarbons]; Apparatus and process for treating — in the gaseous phase.* C. S. Davis, Assr. to M. C. Ernsberger. U.S.P. 1,369,787–8, 1.3.21. Appl., 30.4 and 13.7.17.

THE material passes through an atomiser into the lower end of a vertical retort maintained at a "temperature of dissociation," and containing a drum device spaced from the sides of the retort to provide an annular chamber through which the vapours pass to the outlet. Means are provided "to stir the vapours" as they pass through the retort. —L. A. C.

*Hydrocarbons; Art of cracking* — J. W. Coast, jun., Assr. to The Process Co. U.S.P. 1,370,881, 8.3.21. Appl., 27.9.17.

A body of oil is heated under pressure in a horizontal still heated at the bottom, and the vapour is condensed. Oil from the bottom of the still is passed through a filter to remove coke-forming matter, and then returned to the hulk of the liquid, the process taking place within the still to prevent cooling of the oil. —L. A. C.

*Oils; Cracking* — W. O. Snelling. U.S.P. 1,371,268, 15.3.21. Appl., 15.10.13.

THE vapour generated by a body of oil maintained at a cracking temperature is kept in contact with the oil and at one time during the period of treatment is subjected to external pressure to cause compression and condensation of a portion of the vapour. —L. A. C.

*Gas-producer.* W. Clinie, Assr. to Chapman Engineering Co. Reissue 15,057, 8.3.21, of U.S.P. 1,040,148, 1.10.12. Appl., 12.4.20.

SEE F.P. 434,027 of 1911; J., 1912, 220.

*Gas analysing apparatus.* E.P. 106,265. See XXIII.

## II.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Distillation furnaces.* G. Heeley. E.P. 159,908, 18.9.19.

THE vertical combustion flues of the furnace are arranged between the distillation chambers and a regenerator is situated below the latter. The combustion gas flues and air flues of the regenerator are each situated between two waste gas flues, all the regenerator flues being arranged perpendicularly to the length of the distillation chamber. In this way each of them communicates with all the combustion flues, whereby the whole regenerator is kept working continuously, even when one or more of the distillation chambers are wholly shut down. —A. G.

*Decolorising carbon; Preparation of an active* — Zellstoff-fabr. Waldhof. G.P. 330,942, 25.12.19. Addn. to 309,155 (J., 1920, 479 A).

THE residues from the hydrolysis of cellulose are extracted, before carbonisation, with solvents which dissolve resins, e.g., alkaline solutions, hydrocarbons, alcohols, or ketones. —L. A. C.

*Pyroligneous liquids from the carbonisation of wood; Treatment of* — E. A. Barbet. U.S.P. 1,371,461, 15.3.21. Appl., 14.10.18.

IN the destructive distillation of woody material the tar is continuously removed from the vapours whilst these are at a temperature above the condensation point of acetic acid at the existing pressure. The remaining gases are cooled to condense the pyroligneous acid and are scrubbed to remove the last of the methyl alcohol and to leave a combustible gas suitable for use as a fuel. The wash liquor and crude pyroligneous acid are heated to obtain methyl alcohol vapour, which vapour is washed with an alkali to remove acids therefrom, and then again condensed. The crude acid liquor from which the methyl alcohol has been distilled is continuously boiled, the vapours being brought into contact with alkaline liquor to produce an acetate liquor which is passed through a water-insoluble liquid capable of dissolving tarry matter therefrom. —A. G.

*Arc lamp electrodes.* Planawerke A.-G. für Kohlenfabrikation. E.P. 16,364, 19.11.15. Conv., 31.12.14.

PASSAGES or apertures are provided in arc lamp electrodes into which beads formed by the fusion of salts or metals incorporated in the electrodes for the purpose of enhancing the light-giving power of the arc, are drawn and retained by capillary forces, whereby the salts may be completely utilised and the formation of beads or slag upon the crater prevented. —J. S. G. T.

*Vacuum tube.* Ges. für drahtlose Telegraphie. G.P. 331,414, 22.9.18.

THE electrodes are composed of an alloy of iron and nickel, chromium, or cobalt, melted *in vacuo*. Such alloys are more easily worked and are stronger than chemically pure iron at the high temperatures at which the tube is operated. —J. S. G. T.

*Incandescence gas mantles.* South Metropolitan Gas Co., and C. J. D. Gair. E.P. 160,040, 15.1.20.

AN incandescence gas mantle yielding a light more closely approximating to daylight than that afforded by the usual type of mantle is prepared by using a mixture of substantially pure thoria with 0.2–0.4% of ceria. —J. S. G. T.

*Pyroligneous liquids; Process for removing the tar from* — E. A. Barbet. U.S.P. 1,371,460, 15.3.21. Appl., 14.10.18.

SEE E.P. 120,558 of 1918; J., 1920, 57 A.

*Tungsten-silicon alloy.* E.P. 160,373. See X.

## III.—TAR AND TAR PRODUCTS.

*Low-temperature coal tar and its fractions; Oxidation of — under pressure.* F. Fischer and U. Ehrhardt. Ges. Abhandl. Kennt. Kohle, 1919, 4, 230–292. Chem. Zentr., 1921, 92, II., 422–423.

LOW-TEMPERATURE coal tar and also the fraction distilling below 270° C. were subjected to pressure oxidation in a continuous stream of air at 150° C. and 40 atm. pressure. The results left it uncertain whether the hydrocarbons as well as the phenols were oxidised. The product was a black lustrous pitchy

product partly soluble in benzene. The oxidation product of the hydrocarbons separately treated contained a clear, limpid oil and also a reddish-yellow oil which deposited solids when cooled, and dried in air like varnish. The hydrocarbons were thus attacked, although to a lesser extent than the phenols. In an experiment on the oxidation of the tar fraction up to 270° C., the hydrocarbons were scarcely attacked at 100°, whereas the phenols were extensively changed into resins and acids. Lead oxide was found to have no catalytic effect, and the experiment throws light on the behaviour of lead phenolate paints. The lead phenolate, partly by hydrolysis and partly by the action of carbon dioxide, is slowly decomposed, forming free phenol, which is slowly oxidised. It seems probable that the oxidation products of low-temperature tar are suitable for use as paints and varnishes.—H. J. H.

*Lignite tar oils; Pressure oxidation of* — F. Fischer and W. Schneider. Ges. Abhandl. Kennt. Kohle, 1918, 4, 143—162. Chem. Zentr., 1921, 92, II., 375.

LIGNITE tar oils when treated with air under pressure are appreciably oxidised at and above 100°, but not at 50° C. The product is partly a tough solid mass and partly an oil, which when distilled in steam yields an almost colourless and odourless distillate changing little in appearance on standing in air. If the oxidation is conducted in presence of alkalis scarcely any of the black pitchy material otherwise formed, is produced, but salts are formed from which the acids can be precipitated as viscous liquids with a pungent smell. These acids are insoluble in petroleum ether and have a sp. gr. greater than 1. The acid products melt with decomposition at 135° and resemble the polynaphthenic acids described by Charitschkoff.

—H. J. H.

*Lignite tar oils; Possibility of purifying* — by pressure oxidation. F. Fischer and W. Schneider. Ges. Abhandl. Kennt. Kohle, 1918, 4, 163—179. Chem. Zentr., 1921, 92, II., 375.

THE purifying effect of pressure oxidation on lignite tar oils (*cf. supra*) is compared with that due to alkali and acid washing. By oxidation with air at 35 atm. and 200° C. for 14 hrs. the acid content of an oil was reduced from 10% to 3–4%, and a little additional washing sufficed to give a product similar in outward appearance to that obtained by Gluud (*cf. J.*, 1920, 223 A) in the normal manner, but apparently still containing neutral oxygen compounds. The presence of lime favoured the purification process.—H. J. H.

*Montan wax; Proportion of* — in low-temperature lignite tar and low-temperature tar paraffins. W. Schneider and O. Jantsch. Ges. Abhandl. Kennt. Kohle, 1919, 4, 363—372. Chem. Zentr., 1921, 92, II., 423.

THE montan wax content of several low-temperature tar products derived from lignite was determined, using the insolubility of the wax in cold petroleum ether as compared with corresponding paraffins to effect a separation. The tar was first separated from free carbon by extraction with hot benzol, which was then evaporated off. 20 g. of the residue was extracted successively with 20 c.c., 10 c.c., and 10 c.c. of cold petroleum ether. The residue from a low-temperature tar from lignite was 38%, from Union briquettes 55%. The crude paraffin obtained by distillation in superheated steam of various lignite low-temperature tars was examined similarly. That from lignite yielded 10% of crude montan wax, that from Union briquettes 24%, and from the lignite tar 3%. The authors believe that during the steam distillation loss of montan wax occurs by decomposition.—H. J. H.

*Hydrocarbons; Pressure oxidation of aromatic* — H. Schrader. Ges. Abhandl. Kennt. Kohle, 1919, 4, 310—341. Chem. Zentr., 1921, 92, I., 537.

THE auto-oxidation of aromatic hydrocarbons under high pressure by means of air in presence of aqueous alkali takes place at comparatively low temperatures (200° C.) so rapidly that in a few hours considerable quantities of oxidation products are formed. The oxidisability increases in the order, benzene, naphthalene, anthracene, and, with benzene homologues, with the number of aliphatic groups. Benzene and chlorobenzene first show signs of oxidation at 260° C.; at this temperature it is very considerable with naphthalene, phthalic acid being formed, whilst anthracene is distinctly oxidised at 210° C., anthraquinone being the chief product. With the benzene homologues the oxidation results in the conversion of some or all of the side chains into carboxyl groups. In addition to the above-named products all the hydrocarbons suffer degradation in varying extents to oxalic, formic, and acetic acids and carbon dioxide. Generally the presence of aldehydes could be established as intermediate products. The mechanism of these reactions, and the bearing of auto-oxidation on the processes occurring when aromatic hydrocarbons are used as fuel in an internal combustion engine, are discussed.—G. F. M.

*Catalytic hydrogenation.* Sabatier and Kubota. See XX.

#### PATENTS.

*Mineral tars other than coal tar; Process of treating* — W. Anderson and J. Meikle. E.P. 159,802, 31.10.19.

MINERAL tars (other than coal tar), or mixtures of coal tar pitch with a mineral oil or tar oil, are treated with superheated steam as described for the treatment of coal tar (E.P. 158,337; *J.*, 1921, 209 A). Crude Mexican petroleum tar on treatment between 450° and 500° C. yields bitumen, m.p. 180° C., an oil separable into a yellow resinous substance, a middle oil of sp. gr. 0.910, and a light oil of sp. gr. 0.880, and a gas. By employing a higher temperature, carbon and a resinous substance similar to that produced from coal tar are obtained.—L. A. C.

*Pyridine bases; Manufacture of* — Farbwerk. Meister, Lucius, und Brünig. E.P. 147,000, 6.7.20. Conv., 30.3.18. Addn. to 146,869 (*J.*, 1921, 5 A).

A CONSIDERABLE improvement in the yield of pyridine bases and an acceleration of the reaction is realised by heating paraldehyde with aqueous ammonia in presence of an ammonium salt, such as the chloride or acetate. For example, 120 pts. of paraldehyde, 230 pts. of 5.5% aqueous ammonia, and 20 pts. of ammonium chloride, heated at 180° C. for 8 hrs., give a 70% yield of 2-methyl-5-ethylpyridine, b.p. 176° C.—G. F. M.

*Phenols and "other aromatic hydrocarbons"; Process of removing* — from water or other liquids. R. M. Leggett, Assr. to Ozone Co. of America. U.S.P. 1,341,913, 1.6.20. Appl., 23.12.18.

THE liquid containing phenol is treated with alum, or with ferrous sulphate and lime, and after settling and removal of the deposit is treated with ozone.—W. H. C.

*Benzol; Production of rectified and pure products from crude* — H. Heinemann and B. Hellmann. G.P. 329,962, 15.7.16.

THE washed products are rectified in a double battery of distillation columns. Portions of the liquid are tapped off from points on the main columns for rectification within different narrow

limits in the secondary columns. Alternatively the pairs of columns—main and secondary—are coupled in series. Thus the liquid can be treated continuously, although the different pure products boiling within small limits are being simultaneously obtained.—H. J. H.

*Tetrahydronaphthalene; Process for the preparation of —.* A.-G. für Anilinfabr. G.P. (A) 298,541, 2.8.16, (B) 298,553, 13.9.16, and (C) 301,275, 19.1.17.

A mixture of naphthalene vapour and hydrogen is passed, without pressure, over a catalyst consisting of (A) nickel and copper oxides, (B) manganese oxide alone or mixed with nickel and copper oxides, or (C) an oxide of the rare earths, e.g., thoria or ceria, either alone or mixed with one or more of the oxides claimed in (A) and (B).—A. R. P.

*Paint or varnish.* U.S.P. 1,370,195. See XIII.

*Reduction of organic compounds.* G.P. 330,812. See XX.

#### IV.—COLOURING MATTERS AND DYES.

##### PATENTS.

*Cymene [azo] dyestuff and process of making same.* Cymene [azo] dye. C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,921-2, 2.9.19. Appl. 14 and 19.9.17.

(A) MONOAMINOCYME is diazotised and coupled in alkaline solution with an aromatic hydroxy-compound, e.g.,  $\beta$ -naphthol, which gives a bright orange-red dye. (B) Diazotised aminocymene is coupled in acid solution with an aromatic amine of the benzene or naphthalene series, or with an aminosulphonic acid. Cymene-azo-toluylenediamine is a yellow-brown dye.

*DIMETHYL-DI-ISOPROPYLBENZIDINE* (U.S.P. 1,314,924, process of making same. C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,925-6, 2.9.19. Appl. 19 and 15.9.17.

*DIMETHYL-DI-ISOPROPYLBENZIDINE* (U.S.P. 1,314,924, p. 323 A) is tetrazotised and coupled (A) with an aromatic amine or aminosulphonic acid, (B) with an aromatic hydroxy-compound or sulphonic acid thereof.

*Sulphur dyes and process of making same.* Green sulphur dye and process of making same. C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,928-9, 2.9.19. Appl. 27.9.17 and 27.2.18.

(A) A mixture of 5-aminocymene, *m*-toluylenediamine, and sulphur is heated at 200° C. for 5 hrs. and the melt treated with sodium sulphide and water. A yellow dye is obtained. The aminocymene may be replaced by other nitrogen compounds of cymene, and other aromatic nitrogen compounds may also be present. (B) A green sulphide dye is obtained by heating a mixture of acetaminocymene, a *p*-amino-hydroxy-aromatic compound, and sulphur to 200°–300° C.

*Disazo dyes.* W. M. Ralph, Assr. to National Aniline and Chemical Co., Inc. U.S.P. 1,371,979, 15.3.21. Appl. 24.4.20.

THE intermediate obtained by coupling 1-diazo-4-nitronaphthalene-6 (or 7)-monosulphonic acid with *m*-amino-*p*-cresol methyl ether is diazotised and coupled with 2.8.6-aminonaphtholsulphonic acid. The product dyes cotton, wool, silk, and other fibres blue shades which can be developed to give blue-black to green shades.—L. A. C.

*Indigoid dyestuffs; Manufacture of —.* Farbenfabr. vorm. F. Bayer und Co. G.P. 298,098, 14.4.16.

HALOGENATED derivatives of isatin or naphthhisatin capable of reacting in the  $\alpha$ -position are condensed with 1-hydroxy-6-naphthol ethers, or derivatives of the same not substituted in the  $\alpha$ -position to the  $\alpha$ -hydroxyl group. The dyestuffs produced may be further halogenated. Dichloroisatin- $\alpha$ -chloride and dihydro- $\beta$ -naphthhisatin- $\alpha$ -chloride on condensation with 6-methoxy-1-naphthol yield products which dye respectively grey and olive-green shades; the former on halogenation gives a product which dyes black shades. 6-Methoxy-1-naphthol, h.p. 185° C. (12 mm.), is prepared by acetylation of 1-amino-6-naphthol, methylation of the hydroxyl group, hydrolysis of the acetyl group, diazotisation, and subsequent boiling.—L. A. C.

*Disazo dyestuffs for wool; Manufacture of —.* Badische Anilin- und Soda-Fabr. G.P. 330,824, 6.4.19.

TETRAZOTISED 4.4'-diaminobenzophenone is combined with 1 mol. of  $\beta$ -naphthol and 1 mol. of  $\alpha$ -naphtholdisulphonic acid, yielding dyestuffs which dye wool in an acid bath scarlet shades fast to washing, milling, and light.—L. A. C.

*N-Dihydro-1.2.1'.2'-anthraquinoneazine [Indanthrene]; Process for the preparation of chlorinated —.* Farb. vorm. Meister, Lucius, und Brüning. G.P. 331,283, 28.11.16.

N-Dihydro-1.2.1'.2'-anthraquinoneazino is treated with sulphuryl chloride at 50°–100° C. in the presence of nitrobenzene.—A. R. P.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Microscope sections of cotton yarns and fabrics Rapid method for cutting —.* R. S. Willow and A. C. Alexander. J. Text. Inst., 1921, 12, 99–100.

SOLUTIONS of cellulose nitrate (this gives better penetration and does not require protection from moisture during drying) and acetate are suitable for mounting sections of loose fibres, yarns, and fabrics. Two solutions of the acetate are used (a) a viscous syrup in acetone, and (b) 5–6 g. dissolved in 100 c.c. of acetone. The fibres under examination are immersed for 1 hr. in (b). A clear microscopic slide is painted with a layer of (b) nearly dried, two or more layers of (a) are added and on these the sample fibre is placed. More layers of (a) are then added, the slide is dried over calcium chloride so as to obtain a clear film, then hung in acetone vapour until the film becomes soft, and this is removed and embedded in paraffin wax of m.p. 40°–45° C. In order to observe "Ball's growth rings," the section of a coarse cotton yarn is washed with xylol and absolute alcohol, and stuck to the slide with a smear of albumin (white of egg 25 c.c. glycerin 25 c.c., and sodium salicylate 1 g.) without heat. The slide is held in acetone vapour till the dope softens and is then immersed in acetone for 2 hrs. to remove the dope, whereby each fibre stands freely on end on the slide. Caustic soda (45° Tw. (sp. gr. 1.225) is allowed to pass under the cover slip and the growth rings may then be observed.—A. J. H.

*Lignin and the sulphite [pulp] digestion process.* W. Fuchs. Ber., 1921, 54, 484–490.

Up to the present it has only been shown definitely that the lignin substances are not homogeneous and that their complexity is probably analogous to the

of the proteins or carbohydrates of high molecular weight. Phenols are certainly present in them. They contain methoxy, hydroxy, acetyl, and carbonyl groups and also double bonds. Very little is known of the main portion of the lignin molecule and its structural relationships. The manufacture of sulphite pulp includes at least two separate processes, the detachment of the lignin substance from its natural form of combination in the wood and its subsequent conversion, possibly after further transformations, into soluble sulphur compounds. The nature of the former process is quite uncertain, since nothing is known definitely concerning the mode of union or admixture of cellulose and lignin. The second process may depend on the addition of sulphurous acid, at the double bond, to the carbonyl group or to the phenolic hydroxyl group; further, the possibility must be taken into account that the phenolic nuclei may behave in the sulphite process in their tautomeric form and therefore as unsaturated cyclic ketones.—H. W.

*Oxy- and hydro-cellulose; Differentiation between — by titration.* C. G. Schwalbe and E. Becker. Ber., 1921, 54, 545—550.

EXPERIMENTS were made with oxycelluloses prepared from cotton by the action of bleaching powder, permanganate, and hydrogen peroxide, respectively, and from purified cellulose ("cellulose for nitration") by the action of bleaching powder, and a hydrocellulose prepared by the action of 5% sulphuric acid. The air-dried material (1 g.) was mixed with water (about 50 c.c.) and titrated with N/100 sodium hydroxide solution. Litmus or phenolphthalein (at 80° C.) was used as indicator, the results being similar, but the end-point rather sharper with the latter. The titrations did not proceed very smoothly but were complete within a few hours (the acids are very sparingly soluble and only slowly neutralised). With bleaching powder oxycelluloses a very slight alkalinity was observed directly, due to the fact that the acids were largely present as their calcium salts; when the material was incinerated and the alkalinity of the ash added to the observed acidity, the expected values were obtained. It is thus shown that the "total acidity" of the oxycelluloses is very considerably higher than that of the hydrocelluloses or of the original materials. The presence of oxycellulose in over-bleached cellulose was readily demonstrated in a similar manner. Hydro- and oxycelluloses may be readily distinguished qualitatively by mixing the preparations with distilled water and adding a drop of methyl orange. The liquid is coloured yellow in nearly all cases but is reddish orange with certain oxycelluloses. A few c.c. of concentrated sodium chloride solution is then added, which leaves the colour of the cellulose or hydrocellulose suspensions almost or entirely unchanged but gives a wine-red colour to the oxycellulose preparations. The method may be made quantitative by titrating back with N/100 sodium hydroxide solution; methyl red, however, should be used as indicator, since the end-point with methyl orange is indistinct.—H. W.

*Newsprint [stock]; Recovering —.* C. Baskerville and R. Stevenson. J. Ind. Eng. Chem., 1921, 13, 213—214.

TREATMENT with alkali alone is insufficient to remove the ink from waste newspapers; but by addition of fuller's earth, which remains in suspension or in colloidal solution, the oil in the binding agent, and the carbon, are completely separated. The alkali and fuller's earth should be placed in the pulping machine, and heated to 50° C., before introducing the newspaper; the most suitable proportions are 60 lb. of sodium hydroxide, or preferably 200 lb. of soda ash, and 100 lb. of fuller's earth, per ton of paper. Pulping

is complete in 1 hr.; after washing, the product may be bleached for 15 mins. with 20 lb. of sulphur dioxide per ton of newsprint stock.—W. J. W.

*Book [paper] stock; Regenerating —.* C. Baskerville and C. M. Joyce. J. Ind. Eng. Chem., 1921, 13, 214—215.

BORAX, 10 lb.; soap, 10 lb.; kerosene, 2 galls.; and pine oil, 2 galls., are added to 2000 lb. of bookstock, the mixture being pulped for 1 hr. at 75°—90° C. The treated pulp is washed and bleached. (Cf. U.S.P. 1,351,092; J., 1920, 386 A.)—W. J. W.

#### PATENTS.

*Silk and other yarns; Treatment of — to enable them to be worked up into fabrics.* L. Mayfield. E.P. 159,907, 14.6.18.

YARN which has had no previous dressing treatment whatever is wound on spools, bobbins, or pirns, and placed on shelves within a chamber the atmosphere of which is made humid by means of steam jets which impinge on the surface of water contained in a shallow tank secured to the bottom of the chamber. Yarn treated in this manner for at least 24 hrs. loses its harshness and may be easily worked up into fabrics on knitting machines.—A. J. H.

*Fibres for spinning, e.g., typha fibres, which have deteriorated or have been produced in a porous or rough state; Improving —.* F. Dannert. G.P. 310,763, 16.8.18.

THE fibres are soaked in a bath containing, e.g., 0.2% of sodium-albumin, casein, or egg albumin in solution, and are subsequently treated with formaldehyde or a fixing salt.—L. A. C.

*Fibres; Production of textile — from wood, straw, or the like.* R. Steimmig. G.P. 330,283, 12.8.17.

THE wood or straw is arranged so that the fibres are parallel to one another, and the material binding the fibres together is dissolved by prolonged treatment under pressure with aqueous sodium sulphide solution, firstly just below 100° C., then at the boil, and finally at a lower temperature.—L. A. C.

*Textile fibres; Production of — from the fungus *Phycomyces nitens*.* H. Conradi, A. Jentsch, P. Kraus, and P. Nitsche. G.P. 330,579, 29.11.19.

THE sporangium carriers (hyphae) of the fungus are collected, and after suitable treatment are spun either alone or mixed with other fibres.—L. A. C.

*Paper yarns and fabrics; Process for waterproofing —.* H. Th. Böhme A.-G. Chem. Fabr. G.P. 332,473, 12.4.17.

PAPER goods are waterproofed with montan wax applied as a weak alkaline emulsion followed by a solution of a salt of a heavy metal. The viscosity and permanence of the wax emulsion may be increased, and the finish of the goods improved, by the addition of gelatin, starch, or agar-agar.—B. V. S.

*Cellulose boilers and the like; Process for charging —.* Zellstoff-fabrik Waldhof, and A. Schneider. E.P. 147,417, 7.7.20. Conv., 4.4.19.

WOOD, straw, chips, etc., for use in cellulose boilers are first compressed into briquettes, with or without the use of binding substances (such as the concentrated waste liquors from cellulose boilers), whereby a great economy of space is effected.—A. J. H.

*Cellulose; Process for obtaining — from wood and similar material.* E. Schmidt. G.P. 331,907, 18.12.19.

THE material is treated with a solution of chlorine dioxide in water, carbon tetrachloride, chloroform, or other solvent, hot or cold, preferably under

pressure, and then boiled with caustic soda solution. The method is recommended for the further purification of cellulose obtained by other processes.

—B. V. S.

*Nitrocellulose and its compositions; Method for reducing the viscosity of solutions of —.* O. Carlsson and E. Thall. E.P. 136,141, 9.9.19. Conv., 6.12.18.

A SOLUTION of nitrocellulose in any of the usual solvents is heated in an autoclave (so as to avoid escape of solvent) at a temperature exceeding 60° C., but below the temperature at which decomposition takes place. Solutions capable of being easily filtered are obtained, and their viscosity is determined by the temperature and the duration of the heating.—A. J. H.

[Cellulose acetate.] *Plastic masses; Manufacture of —.* H. Dreyfus. E.P. 160,225, 3.12.19.

IN order to obtain uniform plastic masses when cellulose acetate is kneaded with solvents of high boiling point (plastifiers) (cf. E.P. 132,283, 133,353, and 154,334; J., 1919, 896 A; 1920, 14 A; 1921, 42 A), the kneading is carried out in the presence of a quantity of a volatile liquid such as ethyl and methyl alcohols, benzene, etc., which has no solvent action on cellulose acetate, but which dilutes the plastifier so that it remains inactive until the volatile liquid is evaporated off.—A. J. H.

*Cellulose-nitrate composition. Cellulose-ester composition.* H. T. Clarke, Assr. to Eastman Kodak Co. U.S.P. (A) 1,370,878 and (B) 1,370,879, 8.3.21. Appl., 17.3.19.

THE compositions contain a simple dialkyl sulphone and (A) cellulose nitrate or (B) a cellulose ester.

—L. A. C.

*Cellulose articles; Process of treating regenerated —.* A. D. Conley and E. C. Stillwell, Assrs. to J. P. Hooper. U.S.P. 1,371,300, 15.3.21. Appl., 3.6.19.

REGENERATED cellulose articles are subjected to the action of "soluble castor oil," followed by treatment with an alkali hydroxide solution.—W. J. W.

*Vulcanised fibre masses from cellulose; Production of —.* E. Schwabe. G.P. 324,281, 22.10.18.

CELLULOSIC materials, such as paper, cardboard, or textile fabric, are first coated with a porous filling material without the use of any adhesive and then hardened by treatment with a concentrated solution of a metallic salt, especially zinc chloride, followed by compression of the individual layers into a mass.—H. J. H.

*Light, elastic material; Production of a — from chemically hardened cellulose.* Köln-Rottweil A.-G. G.P. 330,204, 14.6.19.

DISINTEGRATED cellulose material (vulcanised fibre) is worked to a plastic mass with regenerated cellulose or cellulose derivatives and/or other binders such as rubber or resin.—L. A. C.

*Printers' ink; Removal of — from printed matter [paper].* Newsprint Reclaiming Corp., Asses. of J. Jespersen. E.P. 138,628, 3.2.20. Conv., 1.7.18.

IN the preparation of pulp from printed paper, the removal of oils not saponifiable by alkalis and present in printers' ink is effected in a beater engine by means of a strainer, consisting of a perforated plate inclined at 45° to the horizontal. The perforations allow oil but no paper pulp to pass through the plate and collect before a barrier placed behind the plate. The oil is continuously withdrawn, and excessive frothing around the barrier is prevented by a spray of water.—A. J. H.

*Paper-making machines.* D. Russell. E.P. 14,660, 18.6.14.

IN a Fourdrinier paper-making machine a stream of hot or cold dry air is directed by means of a distributing pipe against one side of the felt, which at the time is not in contact with the supporting roller or the paper web, and is drawn through the felt by means of a suction pipe on the other side. Felts treated in this manner last longer and run cleaner, and the paper web tends to be flatter.

—A. J. H.

*Paper; Art of making water-resistant —.* W. Hoskins. U.S.P. 1,370,650, 8.3.21. Appl., 9.4.20.

A RELATIVELY small proportion of an insoluble fatty acid is thoroughly incorporated with the pulp.

—A. R. P.

*Paper; Process for sizing —.* F. Hassler. G.P. 331,549, 28.11.15.

THE condensation products obtained by treatment of aromatic hydrocarbons, or of mixtures of these with aromatic sulphonic acids, with formaldehyde, or by treatment of aromatic hydrocarbons with insufficient sulphuric acid for complete sulphonation, or of sulphones, are claimed for use in the tub-sizing of paper. These are added to the paper-pulp in the hollander alternately with the usual resin-precipitant, from ¼% to 5% of the weight of the paper being used according to the degree of sizing required.—B. V. S.

*Sizing paper pulp.* Zellkoll Ges. E.P. 147,002, 6.7.20. Conv., 10.8.16.

SEE G.P. 303,828 of 1916; J., 1920, 264 A.

*Drying machines [for fabrics]; Doll-head bearings for steam cylinders of —.* J. Ogden. E.P. 160,481, 15.10.19.

*Washing wool and other fibrous or textile materials or fabrics; Machines for —.* T. W. Hawkins. E.P. 160,485, 21.10.19.

*Paper, board, textiles, and felted or woven textile materials; Testing [resistance to tearing] of —.* S. D. Wells and R. J. Marx. E.P. 160,619, 21.1.20.

*Drying varnished or other paper or like material; Apparatus for —.* F. Jenner. E.P. 160,718, 31.8.20.

*Filter-elements.* E.P. 134,228. See I.

*Rosin soap emulsions.* U.S.P. 1,370,884. See XIII.

*Rubber-like material.* G.P. 324,944. See XIV.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Dyeing skins for furs.* Desmurs. See XV.

### PATENTS.

*Dyeing and bleaching apparatus.* A. Wioland. E.P. 146,945, 17.6.20. Conv., 7.7.19.

APPARATUS suitable for dyeing or bleaching fibres or yarns wound on bobbins of all shapes or on roller comprises a reservoir for dye-liquor or bleaching solution and two dyeing or bleaching chamber each consisting of two compartments (open at the top for inspection) and containing a perforate hollow shaft upon which the material to be treated is wound. The liquor may be drawn through the material in either direction, and means are provided whereby air may be forced through the material after dyeing.—A. J. H.

*Dyeing apparatus.* F. B. Petrie and J. Boardman. E.P. 159,548, 29.10.19 and 30.6.20.

AN apparatus suitable for dyeing fabrics and loose or spun fibres consists of a dyeing chamber and a reservoir for dye-liquor, connected by means of swivel members acting as valves, which are so controlled that the direction in which the dye liquor circulates through the dyeing chamber may be reversed, irrespective of whether the liquor is momentarily leaving or returning to the reservoir.

—A. J. H.

*Rau goods; Dyeing of* —. Chem. Fabr. Coswig-Anhalt G.m.b.H. G.P. 330,448, 29.10.14. Addn. to 288,687 (J., 1916, 357).

IN dyeing goods on a tannin mordant the perborates or the like are added to the tannin mordant bath, and not to the dye-bath, as in the chief patent.—L. A. C.

*Decomposition products of proteins; Process for the utilisation of* — [in dyeing]. C. Bennert. G.P. 331,234, 25.12.15. Addn. to 330,133 (J., 1921, 256 A).

PROTALBINIC or lysalbinic acids or the like, or the purified or unpurified salts of these acids, are used in dyeing animal fibres with vat dyestuffs. Good results are obtained without deterioration of the fibre. Decomposition products of casein, rice, or gluten may be added, for example, to weakly acid indigo vats.—A. R. F.

*Printing textile fabrics [cotton and silk].* The Calico Printers' Assoc., Ltd., F. Ashton, and G. Nelson. E.P. 159,552, 28.11.19.

WHEN a fabric printed as for battick styles with a wax resist is immersed in a solution of a metallic mordant and then dried, a large proportion of the mordant is fixed on the printed parts. The degree of fixation decreases irregularly towards the interior of the printed pattern. Novel effects are obtained by printing fabrics with a wax-resist, padding with a solution containing chromium, aluminium, iron, or copper salts, drying, removing the wax resist by means of alkaline solutions, and dyeing the fabric with a suitable mordant dyestuff. Before removing the wax resist, the fabric may be dyed with indigo, ice colours, or any dyestuff capable of being resisted with a wax resist. Veined or marbled effects may be obtained by the usual methods.—A. J. H.

*Colouring design; Producing* — on sheet material. R. Sykora. U.S.P. 1,370,070, 1.3.21. Appl., 25.9.20.

WATER and colouring matter are incorporated with a web of sheet material, and while it is still moist, a solution of a dehydrating agent is deposited upon its surface.—A. J. H.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Sulphur dioxide and trioxide; Determination of* — in burner-gases. R. Dieckmann. Papierfabr., 1921, 19, 285—287.

A MODIFICATION of Reich's method in which the iodine is replaced by  $N/10$  alkali and both methyl orange and phenolphthalein are added as indicators. The two changes from red to yellow and from yellow to orange are very sharp. The volume of gas passed up to the first point corresponds to the percentage of  $SO_2 + SO_3$ , the additional volume to effect the second change to that of  $SO_2$  alone. The second titration in Sander's method (J., 1921, 256 A) after saturation with mercuric chloride gives a somewhat indistinct end-point, to which is attributed the fact

that the method gives low  $SO_2$  values. The author's method is convenient, speedy, and avoids the use of expensive reagents; it gives results accurate to about 2%.—C. I.

*Nitric oxide, nitrogen peroxide, and aqueous solutions of nitric acid; Equilibrium between* —. C. L. Burdick and E. S. Freed. J. Amer. Chem. Soc., 1921, 43, 518—530.

IN the absorption of nitrous gases, such as are formed by the oxidation of ammonia or in the arc process, the principal reactions taking place are  $2NO + O_2 = 2NO_2$ , and  $3NO_2 + H_2O = 2HNO_3 + NO$ . The efficiency of absorption in the towers depends on the extent to which the latter reaction goes to completion, and this depends on temperature, concentration of nitric acid in the absorption liquid, and concentration of nitrogen peroxide in the original gas. The equilibrium conditions for this reaction have been determined experimentally for a wide range of conditions, and from the results a table has been constructed showing the percentage absorption of nitrogen peroxide at temperatures from  $10^\circ$  to  $75^\circ$  C., using as absorbing liquid nitric acid from 5% to 65% in strength and using original gases containing nitrogen peroxide varying from 0.1% to 20%. The favourable conditions for efficient absorption are low temperature, dilute nitric acid, and high concentration of nitrogen peroxide in the gas. Nitric acid above 50% in strength is a very poor absorber; in fact, if there is even a small proportion of nitric oxide in the gas, absorption will not occur at all until this has been oxidised by excess of oxygen. (Cf. J.C.S., May.)

—E. H. R.

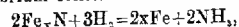
*Phosphoric acid; Concentration of* — by spray evaporation. H. E. La Bour. J. Chem. and Met. Eng., 1921, 24, 466—468.

THE difficulties incidental to the evaporation of phosphoric acid, owing to formation of scale from sludge and metaphosphate deposited on heated surfaces, are avoided by a spraying process in which the acid is heated without reaching its boiling point. The heating tank consists of two concentric compartments, the outer one being provided with a partition. Weak acid is introduced into the outer compartment near one side of this partition and flows round to an outlet near its other side, being heated in circulation by means of steam coils. The hot acid is delivered by a centrifugal pump to a spraying machine in which it is atomised and, in contact with air, is freed from water vapour and fluorine; no sludge is deposited in this machine as it is washed out by the acid, but if it is present in large quantities a settling trap may be introduced. The concentrated acid is returned to the inner compartment of the heating vessel. Through an overflow in this compartment a proportion of the finished acid is constantly discharged, but the bulk of it passes into the outer compartment through a communicating opening and is re-circulated, thus affording a supply of heat for evaporation, and ensuring the complete elimination of fluorine.

—W. J. W.

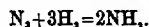
*Iron nitrides; Dissociation pressures of* —. A. A. Noyes and L. B. Smith. J. Amer. Chem. Soc., 1921, 43, 475—481.

THE equilibrium constant of the reaction,



was determined for different mixtures of iron and iron nitride by admitting hydrogen or ammonia into a tube at  $400^\circ$  C. containing iron and iron nitride, and, after allowing sufficient time for equilibrium to be established, withdrawing and analysing the gas. The value of the constant varied with the ratio Fe:N in the solid phase and

indicated the probable existence of a stable nitride  $\text{Fe}_3\text{N}$ , nitrides  $\text{Fe}_2\text{N}$  and  $\text{Fe}_4\text{N}$ , possibly metastable, and a stable nitride  $\text{Fe}_5\text{N}$ . The dissociation pressure of the iron nitride was calculated from  $K_1/K_2$ , where  $K_1$  is the above equilibrium constant and  $K_2$  is the constant for the reaction



The values found for the dissociation pressures were, for the first solid phase, 20,000, for the second, 102,000 to 580,000, and for the third, 41,000 atm. (Cf. J.C.S., May.)—E. H. R.

*Ammoniacal liquor; Distillation of — for the manufacture of liquor ammonia.* W. Wyld. Chem. Age, 1921, 4, 358—360.

An ammonia distillation system designed by the author embodies the following points. Carbon dioxide is removed from the liquor, preheated to 127° C., by spraying it through Körting sprays, at 25 lb. pressure, into an empty column without steam. The ammonia is all retained in solution. In place of a comparatively deep seal to the bubbling hoods, an arrangement of overflowing troughs and an adjustable serrated plate is used, so that the steam merely sweeps the surface of the liquor. In the heat exchanger for condensation of water from the ammonia gas, the inflowing crude liquor is maintained at a definite temperature (being previously heated by the exhausted liquor from the still) to avoid condensation of ammonia. Lime in the form of a thick cream is fed to the fixed ammonia still by gravity. This still is constructed of steel. The apparatus can be worked under a vacuum.—C. I.

*Potassium chlorate; A new crystalline form of —.* E. R. Wolcott. J. Ind. Eng. Chem., 1921, 13, 215—216.

POTASSIUM chlorate is obtained in the form of long, fibrous crystals of silky appearance by treating a saturated solution with an aqueous solution of hydrocarbons prepared from Californian crude petroleum. Equal parts of the latter (20° B.; sp. gr. 1.16) and of oleum are mixed and agitated for 1 hr., without allowing the temperature to rise above the normal. The tar-like product which forms on standing is separated from the acid and dissolved in water to give a solution of sp. gr. 1.066. 10 c.c. of this solution is added to 200 g. of a saturated solution of potassium chlorate and the mixture is diluted to 800 c.c., boiled, filtered, and crystallised. Concentrated sulphuric acid or liquid sulphur dioxide may be used instead of oleum. Although in preliminary tests the crystals produced by the above method appeared to be less sensitive to friction than the ordinary variety, no essential difference was found with the frictional pendulum; but the crystals may lend themselves more readily to coating with a protecting film to reduce sensitiveness. (Cf. U.S.P. 1,355,203; J., 1921, 240 A.)—W. J. W.

*Arsenious compounds; Volumetric determination of — by means of potassium bichromate.* R. Maurice. Ann. Chim. Analyt., 1921, 3, 85—86.

THE solution containing the arsenious compound (e.g., sodium arsenite) is treated with 20 c.c. of 10% potassium bromide solution and its own volume of concentrated hydrochloric acid, and the mixture is titrated with standardised potassium bichromate solution. During the titration, a current of air is bubbled through the mixture and then through a mixture of starch paste and cadmium iodide solution. As soon as all the arsenious oxide has been oxidised, the addition of a further drop of bichromate solution liberates bromine, which is carried over into the iodide mixture, and a blue coloration is obtained.—W. P. S.

*Silicic acid.* V. Lenher. J. Amer. Chem. Soc., 1921, 43, 391—396.

VERY finely ground silica (pure sand ground to particles of about 0.004 mm. diam.) by the prolonged action of water forms a colloidal solution containing about 0.032 g. per l. When heated under pressure with excess of water at 300°—450° C. the same material forms a gel containing 15—18% of water. Attempts to obtain silicic acid or definite hydrates of silica by subjecting silica gels to enormous pressures were unsuccessful. All forms of silica, fused quartz, or quartz crystals, are converted into gels by prolonged treatment with water at temperatures of 300°—600° C. under pressure. It is concluded that the action of water on silica is that of a solvent, forming colloidal solutions or gels, and that the silicic acids are purely hypothetical.—E. H. R.

*Mesothorium and radium; Isotopism of — and separation of these elements from barium.* R. K. Strong. J. Amer. Chem. Soc., 1921, 43, 440—452.

BARIUM chloride containing mesothorium and radium was converted into hydroxide and the radioactive elements were concentrated by fraction crystallisation. The rich hydroxide was then converted into bromide and the radioactive element further concentrated by recrystallisation of the bromide. The bromide method is much more efficient than the hydroxide method for the concentration. The ratio of mesothorium to radium was found to be unaffected by either fractionation process, and it is concluded that the two elements are true isotopes. (Cf. J.C.S., May.)—E. H. R.

*Polythionic chlorides; Existence of — in solution of sulphur in sulphur monochloride.* G. Bru and M. Amadori. Atti R. Accad. Lincei, 1912, I, 217—223.

MIXTURES of sulphur and sulphur monochloride depress the freezing point of bromoform to an extent less than the sum of the depressions produced by sulphur and sulphur monochloride separately, and it is concluded that in the mixture solution polythionic chlorides are present in equilibrium with their components. The tetrathion chloride,  $\text{S}_4\text{Cl}_2$ , is the highest member the presence of which in the bromoform solution is definitely indicated by the abnormal freezing point depression, but it is probable that chlorides richer in sulphur exist in solutions of sulphur in sulphur monochloride. The presence of polythionic chlorides explains the formation of polythio derivatives by the action of ordinary sulphur monochloride on organic substances and the formation of products containing sulphur in excess of the ratio  $\text{S}:\text{Cl}$  in the cold vulcanisation of rubber by means of sulphur monochloride.

*Sodium amide; Preparation and properties of —* J. M. McGee. J. Amer. Chem. Soc., 1921, 43, 586—591.

SODAMIDE melts sharply at 208° C., and has no solvent action on glass below 240° C. when free from sodium hydroxide. At 270°—300° C. a slight action was noticeable after 2—3 days. Above 210° C. it decomposes under the catalytic influence of platinum into ammonia and either sodium imide or nitride. (Cf. J.C.S., May.)—J. K.

*Boron; Preparation of — by the dissociation of boron bromide.* F. Meyer and R. Zappner. Ber., 1921, 54, 550—560.

UNDER the influence of a high-tension arc boron bromide undergoes partial dissociation into boron and bromine, thus giving the most convenient method for preparing the former. Pure boron bromide is prepared by the action of the halogens on crude boron, and is subjected to the arc in the



gaseous form. The action of the arc on liquid boron bromide does not give satisfactory yields. Two forms of apparatus are described, both of which are constructed entirely of glass with ground-in joints. The first is suitable only for small quantities, and consists of a flask in which the bromide is boiled and which is attached to the discharge vessel, which is connected with a reflux condenser. In the second form the boron bromide is vaporised in a flask from which it passes over copper "wool" heated at 200° C. (to remove free bromine), thence through the vessel containing the electrodes, then to the condenser which returns the unattacked bromide to the flask. The boron obtained by this method is a very finely-divided, deep black powder which slowly becomes oxidised on exposure to air.—H. W.

**Boron nitride; Preparation of considerable quantities of pure —.** F. Meyer and R. Zappner. Ber., 1921, 54, 560—566.

THE method consists in the thermal decomposition of the additive compound of ammonia and boron chloride into boron nitride and ammonium chloride. The apparatus consists of a silica tube wound with nichrome wire in three zones. It is fitted at one end with an arrangement for the introduction of regulated amounts of hydrogen, hydrogen charged with boron chloride vapour, and ammonia respectively, and is provided with a platinum pyrometer. The other end is adapted to a wider glass cylinder which serves for the collection of ammonium chloride and which can be heated electrically in such a manner that a uniform deposit of the salt is secured, thus avoiding the otherwise easy choking of the tubes. After the whole apparatus has been dried in a current of hydrogen at 300° C. the tube is heated at 500°—600° C., and regulated streams of ammonia and hydrogen charged with boron chloride are admitted, the former being always in large excess (this procedure ensures the production of a dense additive product). After complete admission of the reagents, the tube is heated in alternate zones for 5—6 hrs. at 500°—600° C. thus avoiding the mechanical removal of the boron nitride in the evolved gases, after which the temperature is gradually raised to 1000° C., whilst a lower current of ammonia passes over the product. As thus prepared (yield 80—85% calculated on the amount of boron chloride used) boron nitride is a colourless powder the density and stability towards water of which depend on the final temperature employed; at 800° C., and with a long period of experiment a very voluminous product is obtained which emits the odour of ammonia after exposure to the atmosphere for some time; more intensely ignited preparations are more stable towards moisture.—H. W.

**Chlorine heptoxide; Preparation of —.** F. Meyer and H. G. Kessler. Ber., 1921, 54, 566—571.

THE preparation of chlorine heptoxide, in substance or dissolved in carbon tetrachloride, from potassium perchlorate and chlorosulphonic acid is described, but the complete elimination of impurities containing sulphur (not exceeding 2% in amount) appears impossible. Pure chlorine heptoxide can be prepared from phosphoric anhydride and perchloric acid (70%), the former being added very cautiously to the latter or, preferably, the components being previously absorbed by or mixed with ignited kieselguhr or "K-silicic acid" (Elektro-Osmose A.-G.). The yields are poor, but the latter method is very suitable when a small quantity of the pure material is required. Pure solutions of chlorine heptoxide in carbon tetrachloride are obtained by cautiously adding perchloric acid (70%) to a suspension of a large quantity of phosphoric oxide in carbon tetrachloride, which is violently agitated and maintained at 0° C.; the product is warmed and filtered, yielding a

solution containing about 2.5% of heptoxide, which can be raised to 7—8% by distilling the solvent as far as possible at 0° C. in a water pump vacuum, digesting the residue for some time at 70°—75° C., and subsequent distillation in a vacuum till a temperature of 80° C. is recorded; small amounts of chlorine and chlorine peroxide were removed by warming the distillate at 80° C. If more concentrated solutions are required, the whole process is repeated, using the 7—8% solution as starting material; three successive treatments give a product containing 20—25% Cl<sub>2</sub>O<sub>7</sub>. (Cf. J.C.S., May.) —H. W.

**Ammonia recovery in sugar manufacture.** Andriik and Skola. See XVII.

**Arsenate and arsenite.** Sears. See XXIII.

**Nitrous fumes in air.** Moir. See XXIII.

#### PATENTS.

**Nitric acid; Method of producing —.** Y. Kawakita. U.S.P. 1,371,789, 15.3.21. Appl., 2.4.18.

By means of a jet of compressed air, a jet of water is drawn into, and atomised in, a chamber containing water. Intermittently, an electric current is passed through the water jet, and electric sparks are passed between it and the water in the chamber, by which the air becomes rapidly heated and nitrogen oxides are formed; the heat generated by the sparks further serves to evaporate the atomised water, and the resulting fall in temperature cools the generated gas, with formation of nitric acid. —W. J. W.

**Nitric acid; Production of concentrated —.** Badische Anilin- und Soda-Fabr. G.P. 302,411, 24.7.17.

A MIXTURE of dilute nitric acid and concentrated sulphuric acid passes down two separate columns in which it is treated with a counter-current of steam. Concentrated nitric acid is delivered from the first column, and a portion of the vapour issuing from the second column, in which the last traces of nitric acid are removed from the mixture, is employed in the first column instead of fresh steam. —L. A. C.

**Nitric acid; Encased column apparatus especially for use in the manufacture of —.** W. Strzoda. G.P. 330,019, 13.10.17. Addn. to 272,153.

THE apparatus, constructed from brittle material cemented and strengthened as described in the chief patent (cf. E.P. 26,732 of 1913; J., 1914, 643), is arranged to provide the maximum reaction surface and area of the gas channels in proportion to the diameter of the tubes. The gas channels, which are of circular cross-section, are arranged around a central overflow tube, and are covered with annular hoods fitted with overflow dishes. —L. A. C.

**Oxides of nitrogen; Process and apparatus for obtaining — from atmospheric air.** F. I. du Pont. U.S.P. 1,370,295, 1.3.21. Appl., 15.7.19.

THE apparatus includes a tubular reaction chamber with a central enlarged portion, a cylinder surrounding the parts near this enlargement, and electrodes in the two ends of the tubular chamber. —C. I.

**Oxides of nitrogen; Process of making —.** A. H. White. U.S.P. 1,370,867, 8.3.21. Appl., 19.6.19.

AN alkaline-earth cyanamide is hydrolysed, and the ammonia liberated is conducted into an absorbent till no more phosphine is generated from the cyanamide; the ammonia, free from phosphine, is then mixed with air and passed over a catalyst. —W. J. W.

*Oxides of nitrogen; Preparation of — by the catalytic oxidation of ammonia.* Badische Anilin- u. Soda-Fabr. G.P. (A) 303,331, 10.12.14, and (B) 307,001, 6.2.16.

BASE metals or their oxides or mixtures or compounds are used as catalysts, and the gas mixture preheated to over 400° C. is allowed to come into contact only with (A) silica or glazed ware high in silica, or (B) oxides or carbonates of calcium, magnesium, or aluminium or a mixture thereof in massive form, before reaching the catalyst.—C. I.

*Nitrous gases; Manufacture of — from the air or other oxygen-nitrogen mixtures by means of the electric arc.* O. Lummer, and Rütgerswerke A.-G. G.P. 331,040, 20.9.18.

THE nitric oxide formed under high pressure in the reaction vessel is cooled *in situ* at a constant rate until it attains a temperature below which it does not dissociate under atmospheric pressure, i.e., 130° C., when it is led into vessels for subsequent treatment.—L. A. C.

*Nitrogen; Regenerative furnace for the combustion of —.* Gewerkschaft des Steinkohlenbergwerks Lotbringen, and M. Kelting. G.P. 331,488, 31.12.19.

TWO rows of narrow jets are arranged on the circumference of a cylindrical reaction chamber, and are connected with a pipe conveying fuel. The jets, which are graded as regards diameter, enter the walls of the chamber at an acute angle, and are provided with several openings arranged in one plane in the form of a fan. The fuel may be gaseous, liquid (as a spray), or solid (as powder). The pointed flames effect a quick mixing of the gases in the reaction chamber.—C. I.

*Nitrogen oxides; Process for the preparation of —.* "Azot" Ges.m.b.H. G.P. 331,591, 27.10.18.

HIGHLY-HEATED nitric oxide or a mixture thereof with air is allowed to expand in a chamber maintained at 150°–300° C., and air or oxygen is admitted. The nitrogen of the air is oxidised along with the nitric oxide. The reaction may be accelerated by a platinum catalyst.—C. I.

*Aluminium nitrate, sulphate, or chloride; Process for the removal of iron from solutions of —.* B. E. D. Kilburn. From Det Norske Aktieselskab for Elektrokem. Ind. Norsk Ind. Hypotekbank. E.P. 139,470, 19.2.20.

IN order to precipitate iron in a readily filterable form 2–10% of the equivalent in calcium carbonate of the aluminium present is added to the solution and a quantity of ferric oxide. The iron is then precipitated with an alkali. The ferric oxide may be washed and used again.—C. I.

*Sea water; Concentration of — [for salt manufacture].* T. A. Reid. E.P. 159,592, 28.11.19.

AN apparatus with which salt can be profitably manufactured from sea water in temperate climates comprises a salt pan in three parts, and either provided with its own fireplace or heated by flue gases from a boiler. In the front part sea water is warmed to at least 26° C., and it then passes to the back part, which is fitted with a series of wooden towers down which the warm sea water flows until concentrated to a sp. gr. of 1.2. It then passes to the central pan, in which the deposition of salt takes place. If a boiler is in use, it may be fed with sea water, which passes on to the evaporating pan when concentrated, and the exhaust steam used for heating another pan.—C. I.

*Catalytic iron [for ammonia synthesis]; Manufacture of — or catalysts containing catalytic iron.* The Nitro-Fixation Synd., Ltd., and H. C. Jenkins. E.P. 159,960, 5.12.19.

A SOLUTION of commercially pure ferric nitrate is freed from phosphorus and arsenic, then from chloride by addition of its equivalent of silver nitrate, and is then evaporated and the residue heated. Before nitrous fumes cease to be given off the heating is stopped and the mass lixiviated and washed. The washings carry away, along with part of the nitrate, the sulphur impurities. The mass is then ignited and reduced by hydrogen or preferably ammonia. If desired, pumice or asbestos freed from impurities may be added to the chloride-free solution. Such promoters as ammonium molybdate, potassium osmate, titanium or uranium nitrate, or an alkali tungstate may be added to the ferric oxide before reduction.—C. I.

*Ammonia synthesis catalyst, and method of making same.* J. C. Clancy, Assr. to The Nitrogen Corp. U.S.P. 1,352,177, 7.9.20. Appl., 25.6.18.

A NUMBER of processes for producing porous cyanamide catalysts at comparatively low temperatures are described. For example, calcium cyanide is obtained by passing hydrogen cyanide into a solution of calcium in liquefied ammonia, and calcium cyanamide by adding calcium to a solution of cyanamide in liquefied ammonia.

*Chemical syntheses [ammonia formation]; Apparatus for effecting exothermic — at high temperature and pressure.* G. Claude, Assr. to Soc. l'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude. U.S.P. 1,371,299, 15.3.21. Appl., 2.2.20.

THE apparatus comprises an outer resisting wall separated by a heat-insulating medium from an inner wall forming a reaction chamber, and having gas outlets. Through this chamber passes an axial tube through which the reacting gases are introduced. Projections on this tube act as supports for catalytic material, and means are provided for delivering an electric current to the tube whereby it can serve as a heating element for the reaction.—W. J. W.

*Nitrogenous compounds; Process of and means for forming —.* J. C. Clancy, Assr. to The Nitrogen Corp. U.S.P. 1,352,176, 7.9.20. Appl., 1.9.17.

BARium cyanide is heated in presence of nitrogen at 400°–500° C. to produce a porous cyanamide especially suitable for use as a catalyst, e.g., in the preparation of hydrocyanic acid from a mixture of hydrogen, nitrogen, and methane or acetylene.

*Carbon bisulphide; Method of making —.* K. P. McElroy. U.S.P. 1,369,825, 1.3.21. Appl., 27.5.19.

FUEL and sulphur are charged into a shaft similar to a gas-producer with an air inlet at the base, and the gases generated are scrubbed with oil to recover carbon bisulphide.—C. I.

*Borax; Process of recovering — from saline waters.* G. B. Burnham. U.S.P. 1,370,273, 1.3.21. Appl., 30.6.19.

THE borax is separated from chlorides, sulphates, and carbonates of sodium and potassium by fractional crystallisation.—C. I.

*Alkali sulphates, alkaline-earth oxides, and hydrochloric acid; Manufacture of —.* Salzwerk Heilbronn A.-G., G. Kassel, and T. Lichtenberger. G.P. 302,496, 9.6.16. Addn. to 299,775 (J., 1920, 749 A).

MIXTURES of naturally-occurring salts of alkalis and alkaline-earths, or of magnesium, containing

chlorides with or without sulphates, e.g., mixtures of carnallite with kainite, or with polyhalite, are melted and treated with steam.—W. J. W.

*Alumina; Treatment of clays for production of —.* P. Askenasy and V. Gerber. G.P. 306,355, 28.3.17.

ALUMINA is obtained from clays and similar materials by heating them at a moderate temperature with barium compounds and lime, in presence of a fluxing agent, such as sodium chloride. The barium aluminate solution is then treated with alkali carbonates or sulphates, and the precipitated barium compounds may be re-utilised in the process.—W. J. W.

*Alumina; Preparation of — from silicious clays.* Aktieselskabet Høyangfaldene Norsk Aluminium Co. G.P. 332,389, 5.7.19.

THE clay is heated with calcium carbonate and a quantity of an alkali compound insufficient to convert the aluminium content of the clay into alkali aluminate, and the product is washed with sodium carbonate solution in two stages.—C. I.

*Sulphur dioxide; Production of — from calcium sulphate.* Chem. Fabr. vorm. Weiler-ter Meer. G.P. 307,772, 20.10.17.

A MIXTURE of calcium sulphate and calcium sulphide is heated above 1000° C., whereby sulphur dioxide is formed in accordance with the equation:  $3\text{CaSO}_4 + \text{CaS} = 4\text{CaO} + 4\text{SO}_2$ .—L. A. C.

*Bleaching powder; Apparatus for manufacture of —.* A. Pfülf. G.P. 329,844, 25.7.19.

To promote the interaction between lime and chlorine, the former is introduced into a shaft-shaped chamber in which are a number of devices, consisting of hollow or solid cylinders, prisms, or rods of star-shaped cross-section, which are mounted on parallel axes and can be rotated. Slot-shaped openings are formed between these devices, through which the material is caused to pass from one side of the chamber to the other.—W. J. W.

*Alkali hypochlorites; Preparation of stable solid solutions of —.* R. Mandelbaum. G.P. 330,192, 11.1.19.

NON-CRYSTALLINE solid solutions of alkali hypochlorites are obtained by mixing them with colloids, or mixtures of these with water-absorptive agents. Thus, sodium silicate solution (38° B.; sp. gr. 1.357), 500 pts., is evaporated to about two-thirds of its volume, and the cooled semi-solid solution is added to 10 pts. of sodium hypochlorite solution (35° B.; sp. gr. 1.320), with or without addition of calcined sodium carbonate.—W. J. W.

*Perborates; Manufacture of — with maximum oxygen content.* Aschkenasi. G.P. 329,845, 27.11.19.

In the manufacture of perborates by any suitable method, the oxidation is not carried beyond a stage corresponding to 23% of active oxygen. In perborates containing 23.4% of available oxygen, the amount is reduced to 21.5% within a year.—W. J. W.

*Crude calcium cyanamide; Manufacture of — in a rotary furnace.* G. Polysius. G.P. 329,961, 18.10.19.

ADHERENCE of material to the walls of the rotary furnace is overcome by cooling the walls, e.g., with water. The most effective procedure is to cause the cooling material to strike the walls obliquely at that part where the material is banked up.—W. J. W.

*Primary alkali phosphates; Manufacture of —.* Farbenfabr. vorm. F. Bayer und Co. G.P. 330,342 and 330,343, 11.9.17.

DISODIUM phosphate is decomposed with hydrochloric acid, the solution is concentrated and separated from the alkali chloride, heated to expel hydrogen chloride, and a secondary phosphate then added to combine with the free phosphoric acid. The mixture of 96% of dihydrogen-sodium phosphate and 4% of sodium chloride obtained may be used for enriching animal fodder.—W. J. W.

*Chlorates, bromates, and iodates; Preparation of —.* H. Schulz. G.P. 330,941, 11.3.19. Addn. to 328,211 (J., 1921, 147 A).

CHLORINE, bromine, iodine, or their hydrogen compounds mixed with air are introduced with or without pressure into water containing a suspended or dissolved salt and a catalyst. By leading hydrochloric acid and air into a suspension of calcium carbonate and manganese dioxide, over 95% of the chlorine is obtained as calcium chlorate.—C. I.

*Copper sulphate; Manufacture of — from residues containing copper.* A. Friedlaender. G.P. 331,144, 5.9.18.

THE residues are dissolved in a solution made strongly acid with sulphuric acid in an electrolytic cell, without a diaphragm, in which the residues act as anode. The copper sludge deposited on the cathode is mixed to a paste with the acid from the cell, or with fresh acid, and is treated with hot air and steam.—L. A. C.

*Mechanical sulphate kilns; Apparatus for feeding sulphuric acid to —.* Farbenfabr. vorm. F. Bayer und Co. G.P. 331,238, 31.7.19.

THE acid is introduced through a small vessel attached to the spindle of the agitator and revolving with it.—C. I.

*Sodium sulphate; Preparation of anhydrous —.* A. Hoffmann. G.P. 331,489, 17.5.18.

GLAUBER'S salt or its solution, pure or mixed with other salts, is treated with volatile substances readily soluble in or miscible with water, or with their vapours, at a temperature above the transition point between the hydrated crystallised salt and the anhydrous salt.—C. I.

*Potash manufacture; Process for treating residual liquors from —.* R. Mewes. G.P. 331,886, 8.7.15. Addn. to 305,082 (J., 1920, 404 A).

THE liquor is treated with crude calcium cyanamide. The nitrogen of the latter is converted into ammonia, or ammonium sulphate or chloride, and the metals precipitated as oxide, carbonate, or sulphate.—C. I.

*Potassium nitrate; Process for the production of — from crude calcium cyanamide.* E. Hene and A. van Haaren. G.P. 332,002, 14.4.20.

CRUDE calcium cyanamide, potassium sulphate, nitric acid, and water are stirred together at a temperature below 100° C., preferably 25°–35° C., and the liquor filtered.—C. I.

*Causticisation of soda in the cold; Process for the —.* Schweizerische Sodafabr. G.P. 332,003, 5.6.19.

A MIXTURE of soda solution and limo is treated in a revolving mixer similar to a centrifugal machine, when interaction is speedy and complete without heating, 10 cub. m. of liquor being causticised in 10 mins.—C. I.

*Magnesium chloride liquors; Treatment of* —. W. Esch. G.P. 332,153, 12.6.19.

PART of the magnesium, after addition of magnesium oxide or hydroxide, is obtained as basic carbonate by treatment with ammonium carbonate below 50° C. The remainder is obtained as hydroxide, with recovery of the ammonia or carbon dioxide and ammonia, the liquor being first freed from sulphate by addition of calcium chloride solution.—C. I.

*Colloidal metallic oxides; Process for the electrical preparation of* —. A. Wander A.-G. G.P. 332,200, 4.9.19.

A MAIN circuit with direct or alternating current passes through an electrode of the metal the oxide of which is required, placed in a medium for dispersion, to which a protective colloid may be added. The metal is oxidised electrolytically, and the layer of oxide is dispersed by the discharge of a condenser placed in a circuit parallel to the main circuit. Oxides of copper, nickel, and iron and oxides and hydroxides of lead, zinc, and cadmium are mentioned.—C. I.

*Oxygen and nitrogen; Processes for separating — and apparatus therefor.* M. Zack. E.P. 152,643, 3.9.20. Conv., 15.10.19.

A COOLED gaseous mixture of oxygen and nitrogen at a certain pressure is introduced into a turbo-engine actuated by an electric motor, and there undergoes further cooling through expansion. By the action of electro-magnets attached to a rotating hollow ring, cooled by liquid air, oxygen, being paramagnetic, is drawn from the turbo-engine at its periphery into the ring, while the nitrogen passes through an outlet parallel to the shaft of the turbo-engine into a centripetal turbine, operated by a second electric motor at a different speed from the first, and thence to an outlet. The gaseous mixture freed from part of the nitrogen passes into a second cooled hollow ring, which is also provided with electro-magnets and is rotated by the second electric motor. Periodically the current to the electro-magnets of the first ring is interrupted, and the oxygen passed into the second ring, from which both gaseous and liquid oxygen can be drawn off through two outlets.—W. J. W.

*Nitrogen; Production of* — [from producer gas] suitable for the catalytic manufacture of ammonia. Badische Anilin- und Soda-Fahr. G.P. 306,302, 3.7.15.

PRODUCER gas is burnt with a small excess of oxygen or air, pure hydrogen is added to combine with the excess of oxygen, and carbon dioxide and other impurities are subsequently removed from the gas.—L. A. C.

*Hydrogen manufacture by iron contact process; Utilisation of heat retained by generated hydrogen and excess steam in* —. F. Lang. G.P. 330,800, 7.11.16.

By means of a system of heat exchangers the waste heat from the hydrogen and steam may be utilised for generating further steam, for preheating the boiler feed water, and for removing air from the cooling water.—W. J. W.

*Hydrogen production by the iron contact process; Process and apparatus for utilisation of the waste heat available in* —. F. Lang. G.P. 332,111, 8.11.16. Addn. to 330,800 (cf. supra).

THE heat of the hydrogen and water vapour leaving the converter and also the heat of the gases leaving it during the reduction, washing, heating, and air blast periods are used for generating the steam required for the process or for the heating of boiler

feed water. The apparatus consists of a heat exchanger with suitable inlet connexions and an exhaust for the waste gases or a two-chambered exchanger heated on one side by the hydrogen and on the other by the waste gases.—C. I.

*Sulphur dioxide; Oxidation of — and catalyst therefor.* F. Slama and H. Wolf, Assrs. to General Chemical Co. U.S.P. 1,371,004, 8.3.21. Appl., 9.10.14.

SEE E.P. 23,541 of 1913; J., 1915, 833. It is specified that the catalyst be distributed on a very finely divided carrier, not exceeding 60 microns in diameter.

*Aluminium nitride; Manufacture of* —. V. Gerber. E.P. 139,195, 18.2.20. Conv., 18.2.19. SEE U.S.P. 1,350,149 of 1920; J., 1920, 657 A.

*Cyanamides; Process for the production of* —. L. E. Friderich, Assr. to Soc. F. Gros et Bouchardy. U.S.P. 1,371,616, 15.3.21. Appl., 17.10.18.

SEE E.P. 132,622 of 1918; J., 1919, 818 A.

*Tin oxide and zinc oxide; Production of* —. D. de Ros. U.S.P. 1,372,407, 22.3.21. Appl., 30.10.18. SEE E.P. 142,157 of 1918; J., 1920, 448 A.

*Nitrogen of the atmosphere; Fixation of the* —. R. Pearson. U.S.P. 1,372,541, 22.3.21. Appl., 13.2.19.

SEE E.P. 130,693 of 1918; J., 1919, 718 A.

*Tin salts.* E.P. 159,659. See X.

*Calcining magnesia.* E.P. 160,231. See XI.

## VIII.—GLASS; CERAMICS.

*Kaolins and clays; Shrinkage of — on drying.* A. Bigot. Comptes rend., 1921, 172, 755—758.

A COMPARISON of certain kaolins and clays and an argillaceous sand from different sources shows that the free silica contained in the clays renders the material less plastic. The plasticity is obtained by dividing the colloidal water (water of plasticity, water adsorbed by the colloids present) by the total water absorbed and multiplying this quotient by the shrinkage on drying. Elevation of temperature or of pressure diminishes both the colloidal and the interposed water (water filling the spaces between the particles), but the diminution of the latter is very slight as compared with that of the former.—W. G.

*Kaolins, clays, and bauxites; Variation of volume of — under the influence of heat.* A. Bigot. Comptes rend., 1921, 172, 854—857. (Cf. supra.)

SAMPLES of kaolins, clays, and bauxites were powdered, sieved, mixed with water, and compressed in moulds. The lengths of the samples were measured from 0° C. at each successive 100° up to the temperature at which they showed signs of fusion. The results are shown graphically and indicate that bauxites, kaolins, and clays, which do not contain any free silica, commence to contract below 1000° C. Any clay or bauxite which expands below 1000° C. contains free silica. Clays and kaolins when heated rapidly puff up before reaching their melting-point, but bauxites do not show this puffing. The swelling or puffing up is due to the volatilisation of silica or silicates.—W. G.

*Heat losses of metallurgical furnaces.* Rosin. See X.

## PATENTS.

*Glass melting furnaces; Gas-fired* — S. G. Curd. E.P. 159,559, 20.11.19.

Two gas producers are used and are operated alternately, the gas being produced by passing a mixture of superheated steam (preferably at 800° F. and 10 atm. pressure) and hot air through burning fuel. The gas and hot air are admitted simultaneously to the melting tank, the flow of gas through the furnace being periodically reversed in direction. The steam is generated and superheated by the waste gases, which also preheat the air. The gas is lightly superheated before use.—H. S. H.

*Lehrs or kilns; Annealing* — for glass and other articles. S. and J. E. Laycock. E.P. 160,282, 19.12.19.

An annealing lehr having an internal endless chain conveyor for carrying the material has the gas ports constructed to pass the heating gases into direct contact with the material being treated in the annealing chamber. A regenerative chamber for the gas is placed directly under the furnace part of the lehr, so that the gas is preheated before it enters the lehr and a better pressure of gas is maintained. Waste hot air from round and about the melting furnace is collected and mixed with the gas before it enters the lehr.—H. S. H.

*Glass composition and articles made therefrom*. E. C. Sullivan and W. C. Taylor, Assrs. to The Corning Glass Works. U.S.P. 1,369,988, 1.3.21. Appl., 3.5.19.

GLASS contains silica, soda, and two divalent oxides in the molecular proportions of 100 of silica to at least 20 of alkali and at least 6 of divalent oxides.—H. S. H.

*Glass; Method of and apparatus for removing striae from melted* — S. R. Scholes, L. W. Nicols, and W. F. Kaufman. U.S.P. 1,370,673, 8.3.21. Appl., 8.10.19.

THE formation of striae in molten glass is prevented by agitating the pot and its molten contents at a uniform temperature until complete mixing of the constituents takes place. This is accomplished by providing means for moving the pot and its contents around an axis inclined to the horizontal.—H. S. H.

*Refractory and [electrical] insulating product and process for the manufacture of same*. G. L. Dimitri and J. E. Delaunay. E.P. 142,512, 1.5.20. Conv., 21.11.16.

NATURAL silicate of magnesium and a flux consisting of one or more natural or artificial multiple silicates (e.g., feldspar) are mixed in the powdered state, moulded or compressed, and after complete desiccation cut or fashioned into the required form and baked at about 1450° C.—H. S. H.

*Refractory articles made from china clay and method of manufacture*. The Osmosis Co., Ltd., D. Northall-Laurie, and W. R. Ormandy. E.P. 159,737, 22.1.20.

THE clay is brought into the sol condition by the addition of a suitable electrolyte, the coarser particles are allowed to settle, and the fine clay particles are separated from the suspension by electrophoresis or by precipitating the sol suspension by means of a solution of aluminium sulphate or other agulant. A slip is made from the purified clay and the articles are cast from it. The treated clay will later at a temperature between 1400° and 1500° C., and the use of feldspar is thus avoided. Grog consisting of vitrified china clay may be added to reduce the firing shrinkage. Articles made in this way are more refractory than those prepared from

kaolin and feldspar and resist temperature changes more effectively than porcelain prepared by any known process.—H. S. H.

*Oven or kiln; Gas-fired* — for use in the manufacture of tiles, pottery, and other ware or other purposes. J. H. Marlow. E.P. 159,522, 23.7.19.

THREE heat-distributing conduits extend throughout the length of the oven, and between the central conduit and each of the side conduits wheeled carriers travel. Each conduit communicates with a combustion chamber placed under it and is perforated so as to allow heat and gases to pass into the interior of the oven. The air is heated before entering the combustion chamber.—H. S. H.

*China clay and other plastic materials; Drying of* — C. J. Grace. E.P. 159,525, 23.8.19 and 25.2.20.

THE china clay sludge is pumped into a tubular filter which is connected with a heated tubular drying chamber. The cake of clay is moved from the filter to the drying chamber either intermittently by means of a ram or continuously by conveyor vanes.—H. S. H.

*Glass; Method and apparatus for the manufacture of* — A. Ferguson, Assr. to F. W. Passmore. U.S.P. 1,371,084, 8.3.21. Appl., 7.9.20.

SEE E.P. 154,251 of 1919; J., 1921, 46 A.

*Firing porcelain*. G.P. 331,924. See X.

## IX.—BUILDING MATERIALS.

## PATENTS.

*Cement materials, magnesite, and the like; Method for burning or calcining* — and apparatus therefor. A. Hauenschild. E.P. 159,780, 28.4.20.

THE charge is inserted with the fuel in the form of a continuous vertical column, the mineral being in direct contact with the fuel on all sides and the usual vertical alternations of fuel and mineral being absent. An annular fuel shoot extends downwards some distance into the kiln shaft and prevents the fuel from mixing with the mineral. Air under pressure enters by blast nozzles at the sides of the kiln shaft. A cover on the shoot prevents the hot gases from escaping without permeating the mineral.—H. S. H.

*Gypsum products; Process of ageing calcined* — and product thereof. H. E. Brookby, Assr. to United States Gypsum Co. U.S.P. 1,370,581, 8.3.21. Appl., 8.3.20.

A CALCINED gypsum product comprises a dry powder of unset commercial plaster of Paris with a small amount of water-soluble deliquescent substance intimately incorporated.—H. S. H.

*Plastic composition and process for making the same*. R. G. Erwin, Assr. to International BitumConcrete Co. U.S.P. 1,370,637, 8.3.21. Appl., 6.7.20.

A PLASTIC composition is produced by suspending a mixture of mineral dust (all of which will pass a 200-mesh sieve, 60% being smaller than 0.02 mm.), sodium chloride, and sodium sulphate in preheated bitumen, and adding pulverised sulphur to the mass. The whole is agitated in a vat subjected to suction and heated sufficiently to combine the ingredients and their derivatives. The composition is then mixed with comminuted vegetable particles of various sizes in sufficient quantity to fill the pores within and the voids between the vegetable particles and to coat and bind them together, and the mass is agitated in a vat under the influence of

sustained heat and suction. After removing the composition from the vat it is compacted to shape.  
—H. S. H.

*Keene's cement; Manufacture of* — W. Hoskins, Assr. to The Best Bros.' Keene's Cement Co. U.S.P. 1,370,968, 8.3.21. Appl., 13.5.18.

*Gypsum* is ground very fine and passed continuously at a uniform rate through a rotary, internally-fired kiln, after which it is mixed with sufficient lime to neutralise all acidity.—A. R. P.

*Heat-insulating material and method of making same.* C. H. Bennett, J. F. Palmer, and F. V. Wedlock, Assrs. to Bentex Co. U.S.P. 1,371,016, 8.3.21. Appl., 18.6.20.

A RIGID, cellular, heat-insulating material is made from a "dough" having the following percentage composition by weight:—Asphaltum, 27.0; infusorial earth, 11.0; magnesium carbonate, 5.0; crude rubber, 26.9; sulphur, 14.9; sulphur-treated corn (maize) oil, 2.6; petroleum tailings, 5.8; sodium bicarbonate, 6.0; alum, 0.8. The mixture is heated under pressure to a sufficient temperature and for a sufficient time for vulcanisation. The material is rolled while still hot and finally cooled. It maintains its rigidity at the ordinary temperatures of artificial refrigeration.—H. S. H.

*Mortar-forming material.* F. Hartner. U.S.P. 1,371,762, 15.3.21. Appl., 1.8.19.

SEE G.P. 312,239 of 1917; J., 1919, 905 A.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Iron, carbon, and oxygen; Chemical equilibrium between* — A. Matsubara. Trans. Amer. Inst. Min. and Met. Eng., Feb., 1921. 52 pages.

EQUILIBRIUM compositions of the gaseous phase in the system, CO-CO<sub>2</sub>-Fe, were measured at 863° C., 1070° C., and 1175° C. The systems, CO-CO<sub>2</sub>-FeO (Fe) and CO-CO<sub>2</sub>-Fe<sub>3</sub>O<sub>4</sub> (FeO), were studied at several temperatures higher than 700° C., also systems containing iron carbide. At a temperature of about 1200° C., under a pressure of 1 atm., the dissociation pressure of the carboniferous iron phase is equal to that of carbon monoxide, and neither carburisation nor decarburisation occurs even in a current of pure carbon monoxide. Lower temperature or higher pressure values increase the relative pressure of carbon monoxide in the gaseous phase. From the data obtained the existence of oxygen-bearing iron is inferred, and the equilibrium conditions are discussed with relation to case hardening, malleable casting, and blast furnace conditions.  
—C. A. K.

*Iron; Influence of copper on the rate of solution of — in acids.* F. K. Bell and W. A. Patrick. J. Amer. Chem. Soc., 1921, 43, 452–465.

EXPERIMENTS with pure iron-copper alloys containing from 0.1% to 5.0% Cu showed that the copper had a marked retarding effect on the rate of solution of the iron in 28.7% hydrochloric acid, as compared with pure reduced iron. The rate of solution was determined by measuring the rate of evolution of hydrogen from a polished surface of the metal in contact with the acid. Mere contact with copper wire had a marked retarding effect on the rate of solution of pure iron, whilst platinum and silver were without effect. Experiments with 57% sulphuric acid gave erratic results. It is emphasised that no conclusions regarding the behaviour of these alloys with dilute acids can be drawn from these experiments with strong acids. (Cf. J.C.S., May.)  
—E. H. R.

*Nickel steel; Static and dynamic tension tests on* — J. J. Thomas and J. H. Nead. Trans. Amer. Inst. Min. and Met. Eng., Feb., 1921. 12 pages.

AN attempt to determine the relation between static and dynamic tensile tests as measured by the work required to break test specimens slowly in a tensile testing machine, and rapidly, by means of a falling weight. Using a nickel steel, tempered to varying degrees of hardness, the results show that for hard steel the total work of rupture is very low under either a slowly or rapidly applied load. As ductility increases the work of rupture increases, and ductility, as measured by elongation and reduction of area in the ordinary tensile test, is important as an indication of strength as measured by work units. Experiments indicate that steel is in its best condition when quenched and drawn at a temperature slightly lower than its critical point.—C. A. K.

*Ferromanganese; Manufacture of — in the electric furnace.* R. M. Keeney and J. Lonerger. Trans. Amer. Inst. Min. and Met. Eng., Feb. 1921. 36 pages.

COMPARING blast furnace and electric furnace production of ferromanganese, the same recovery (72%) of manganese was made in the electric furnace smelting ore containing 34.8% Mn as in the blast furnace smelting 40% ore. The slag and steel losses were practically the same in each case. Of the same grade of ore the electric furnace would show at least 5% greater recovery than the blast furnace, and at the present price of coke, fuel and power costs balance. In smelting oxide ore better operating results were obtained when any necessary iron was added in the form of manganiferous ore rather than in the form of steel turnings. Among the general operating difficulties encountered with the electric furnace, formation of carbide was the most serious. This tendency was modified by the use of lignite as a reducing agent in preference to anthracite or bituminous coal and also by attempting to produce a slag containing less than 12% Mn. Intermittent charging gave better results than continuous working in so far as output, power, and labour costs were concerned. Magnesite is preferred as a material for furnace bottoms though carbon and firebrick bottoms are in use. A 1100-k.w. furnace smelting 34.8% manganese ore and producing ferromanganese containing 73% Mn, the power consumption was 4457 k.w.-hours and the electrode consumption 125 lb. per short ton.  
—C. A. K.

*Gold; Extraction of — from its ores.* R. Paul. Chem.-Zeit., 1921, 45, 285–287.

FOR regulating the flow of pulp to the amalgam plates so as to ensure removal of heavy antimony sulphide, whilst at the same time preserving sufficient time of contact for amalgamation, a device is installed between the tube-mills and the plates. It consists of a gutter bent twice at right angles, provided with cross-haffles which retard the flow and cause deposition of coarse particles of gold. Near the plates the gutter is expanded to the full width, and at this point an automatic regulator is inserted, which interrupts the flow of pulp to allow of amalgamation and then again accelerates it to admit of the antimony sulphide being carried away. Experiments have shown that ball mills are not very efficient in separating gold from its ore, gold apparently free was found to be enclosed in a thin layer of quartz and contained only 12.5% actual free metal capable of amalgamation; substitution of roller mills or tumbler mills may be advantageous. To reduce loss of amalgam from the plates, a "catcher" consisting of a casting with rim and overflow is placed below the plates. The slime enters by means of a central tube.



any amalgam gradually separates on the bottom of the cup. It is periodically transferred to a cast-steel drum containing a few steel balls, in which it is treated with fresh mercury, and then washed with water, and pressed to recover solid amalgam. —W. J. W.

*Platinum and gold; Microscopical detection of — in the Siegerland "grauwacken."* H. Schneiderhöhn. Metall u. Erz, 1920, 17, 511–514.

THE rock is a medium-grained grey sandstone with a sericite binding material. It contains very thin dark streaks running parallel to each other, which, under the microscope, are seen to be enrichment zones of heavy minerals, mostly zircon and rutile. Sections of the mineral were cut and carefully polished by means of magnesia on a piece of flannel. On viewing the prepared specimens under the microscope in transmitted light a few bright yellow particles of metallic appearance could be seen which readily became grey and rough on adding a small drop of mercury to the specimen, thus indicating gold. Platinum could not be detected with certainty in this manner. 150 g. of the finely powdered rock was then treated with hydrofluoric and sulphuric acids for several days on the water-bath. The residue, under the microscope, showed bluish-white and yellow grains of platinum and gold, which were confirmed microchemically as osmium, rubidium, and thallium chloroplatinates and as thallous chloraurate respectively. —A. R. P.

*inc; Value of — in the ore and the economic basis of its recovery.* Paul. Metall u. Erz, 1920, 17, 439–449, 475–573, 514–519; 1921, 18, 59–66, 123–135.

THE customary formula on which zinc ores are sold is  $V = P(t - 8) - K$ , where  $V$  is the value of 1000 kg. of the ore,  $t$  is the zinc content %,  $P$  is the market price of one unit (=10 kg.) of spelter, and  $K$  is the returning charge. The latter varies with the nature of the ore, and the author shows that it should be higher for roasted ores than for raw blende in a proportion which is greater the higher the sulphur content and the price of spelter and the lower the zinc content of the original ore. With equal returning charge the value of zinc in an ore per unit increases rapidly with the zinc content; on the other hand low-grade ores may become of negative value when the price of zinc is low (cf. J., 1919, 18 A). The above formula is examined from every point of view and reasons are advanced to show that it does not fairly represent the value of an ore. Thus the returning charge is the same for rich and poorer ores, while the proportion of "free" units is relatively smaller with rich ores, and hence there is not such a great proportionate margin for losses, therefore the richer ores are dearer than they should be in relation to the poorer. Again the cost of smelting depends not on the amount of ore smelted, but on the capacity of the furnace, and this varies according to the apparent specific gravity of the charge. No account of this is taken in the formula, but of the value of the sulphur. The author therefore suggests two new formulae, one for roasted and one for unroasted ores. In the case of sulphide ore a 1% loss of zinc on roasting is assumed and the loss in weight of  $(t - 15)\%$ , where  $t$  is the sulphur content of the ore. It is also assumed that 95% of the sulphur removed is recovered as sulphuric acid 60° B. (sp. gr. 1.71). If the cost of roasting is approximately 15 M. per ton of roasted ore, while that of making a ton of sulphuric acid is 8.5 M. and its selling price is 25 M., then the value of a ton (10 kg.) of zinc in the ore in M. is given by equation

$$V = 0.08415(P - E) - (115 - t_s)K / 1000 t_s + (0.56878233 t_s - 24.275) / t_s$$

where  $P$  is the market price of 100 kg. of spelter,  $E$  is the profit on every 100 kg. of zinc recovered,  $K$  is the cost of 1 cub. m. of retort room,  $s$  is the apparent sp. gr. of the ore,  $t_s$  is the zinc and  $t$  is the sulphur content of the ore. With oxidised ores this simplifies to  $0.085(P - E) - K / 8 t_s$ . —A. R. P.

*Zinc; Sources of loss in the distillation of —, and the mechanism of the volatilisation of the metal.* O. Mühlhaeuser. Metall u. Erz, 1921, 18, 1–22, 45–51.

BEGINNING with a new retort, the author carried out a series of zinc distillations from roasted blende and anthracite in a small experimental furnace and tables are given showing the yield of metal and of by-products together with the losses. The loss of zinc in the low-grade residues was about 3% and remained practically constant throughout the tests. The loss due to escape of zinc vapours from the receiver was comparatively high with the first charges, but eventually, after 10–12 distillations had been carried out in the same retort, sank to an approximately constant figure of 1.2–1.6%. The proportion of zinc that escaped through the pores and minute cracks in the retort was about 2–3% with a new retort, but after several distillations this loss from this source fell to 0.8–1.2%. The most serious loss of zinc with new retorts appears to be that due to combination of the zinc oxide with the material of the retort with the formation of zinc spinel. This loss may be as high as 25% or more of the total zinc in the charge, but on continued use of the retort it falls to an average of a little over 1%. The total average loss for the first eight charges was about 19% and for the next ten 7%. The mechanism of the volatilisation of zinc is discussed and the need of research to reduce the losses is emphasised. —A. R. P.

*Brasses; Tensile strength of various — at high temperatures.* F. Doerincel and J. Trockels. Int. Zeits. Metallogr., 1920, 12, 340–358. Chem. Zentr., 1921, 92, II., 553.

A NUMBER of tests were carried out on brass cylinders, 18 mm. diam. and 36 mm. high, in a 40-ton testing machine at various temperatures up to 800° C. The test pieces were heated by enclosing them in a resistance tube-furnace and the temperatures were measured by means of a thermocouple and, after testing, calorimetrically. The tensile strength in general falls with rising temperature, and at 800° C. has a value only  $\frac{1}{4}$  that obtained at 500° C.; only with a 58% Cu brass is a minimum value reached at 700° C. The maximum value of both the hardness and tensile strengths of the copper-zinc series is obtained in the region corresponding to the  $\alpha$ -mixed crystal phase, i.e. with alloys containing about 85% Cu. Alloys containing 67–72% Cu exhibit irregularities, which seems to show that the  $\alpha$ -brasses require further investigation. —A. R. P.

*Tin ore; Electric smelting of —.* H. Krebs. Metall u. Erz, 1921, 18, 77–78.

TIN ores become conductors of electricity at moderate temperatures, so that by using large pieces of ore as a resistance, sufficient heat can be developed to melt it and reduce it to metal on the addition of coal. It is not generally necessary to add any slagging material, as the ore usually is self-fluxing, nor is it necessary to protect the tin oxides from going into the slag. It is preferable to carry out the process in a trough furnace with a low charging shaft and two or more electrodes dipping into the bath. Ore and coal are charged in as required and the metal is tapped from the bottom until the hearth is nearly full of slag, which should be rich in tin. This is cleaned by adding a charge of coal only to separate its tin contents as



metal. In this way very pure metal may be obtained and the tin content of the slag reduced to as low as 0.3%, while the losses by volatilisation are much less than by the reverberatory or shaft furnace process.—A. R. P.

*Copper-antimony system; The compounds  $\text{Cu}_3\text{Sb}_2$ ,  $\text{Cu}_2\text{Sb}$ , and  $\text{Cu}_3\text{Sb}$  in the —.* H. Reimann. Z. Metallk., 1920, 12, 321–331. Chem. Zentr., 1921, 92, I., 484.

THE work of Carpenter (J., 1913, 979) brought about changes in the copper-antimony equilibrium diagram, but some points remained to be cleared up. The present investigation has been confined to alloys containing 25 to 33 atoms % Sb. The maximum of the melting-point curve lies not at 25 atoms % Sb, corresponding with a compound  $\text{Cu}_3\text{Sb}$ , but at 29 atoms % Sb or the compound  $\text{Cu}_3\text{Sb}_2$  (m.p.  $632^\circ\text{C}$ ). This compound forms mixed crystals with copper or antimony over the range from 18 to 33 atoms % Sb; the saturated mixed crystals with 33 atoms % Sb undergo decomposition and form a eutectoid at 27 atoms % Sb ( $430^\circ\text{C}$ ).—E. H. R.

*Bearing metals and their technological evaluation.* J. Czochralski. Int. Zeits. Metallurg., 1920, 12, 371–393. Chem. Zentr., 1921, 92, II., 552–553.

THE mechanical properties and uses of various bearing metals are discussed and compared, together with the technique of casting and testing the finished alloys. Copper-tin bearing metals with about 10% Sn consist of a single constituent,  $\alpha\text{-CuSn}$ , which contains generally all the other components of the alloy in solid solution. As the suitability of an alloy for use as a bearing metal depends on its heterogeneous structure, copper-tin alloys that have been rendered homogeneous are not of any use as bearing metals. The utility of a copper alloy as bearing metal depends on the gradual wearing away of the unevenness of structure, whereas that of a tin bearing metal (white metal) depends on the flexibility of the ground mass. Copper alloys are more suitable for bearings that have to withstand a considerable pressure, while lead alloys are preferable if the pressure on the bearings is small. Tin alloys occupy an intermediate position, but they are better than those containing calcium or barium instead of tin. These latter are difficult to prepare on account of the readiness with which they oxidise. It is recommended to cast these alloys at a temperature of  $200^\circ\text{C}$ .—A. R. P.

*Heusler's alloys. II. Magnetic measurements of Heusler's aluminium-manganese bronzes of high iron content.* O. von Auwers. Ann. Physik., 1920, 63, 867–899. Chem. Zentr., 1921, 92, I., 485. (Of. J., 1920, 27 A.)

ADDITION of iron, from 13.5 to 18%, to aluminium-manganese-bronzes does not appreciably affect their magnetic behaviour. It is possible that the manganese content is more important than the iron content. At low temperatures the magnetisability of the Fe-Al-Mn-bronzes generally increases. With a bronze containing 9.2% Al, 17.8% Mn, 13.5% Fe, and 59.6% Cu, the opposite effect was observed at  $-190^\circ\text{C}$ , but not at  $-78^\circ\text{C}$ .—E. H. R.

*Dental amalgam; Volume changes in a — as a result of a transformation at  $70^\circ\text{C}$ .* A. W. Gray. Phys. Rev., 1920, 15, 524–525. Chem. Zentr., 1921, 92, II., 552.

ON heating dental amalgams that had been allowed to harden to their transformation points, those containing several per cent. of zinc rapidly formed crystals, while those free from zinc showed no change. Freshly prepared amalgams, on the other hand, showed the crystal formation in all cases. The crystals consisted of hexagonal plates belonging

to the hexagonal or orthorhombic system. The temperature-volume curve for the zinc-free alloys rises in a straight line to a maximum at the transformation point, rapidly falls to a minimum, and then rises sharply for a short distance, after which a further fall takes place. On re-heating a similar curve is obtained, except that the change point is not so strongly marked, especially with alloys with a low silver and high copper content. Amalgams that contain zinc give a curved line up to the change point, after which the line rises rapidly. This rapid expansion ceases after several hours or if the temperature is kept constant or allowed to fall.

—A. R. P.

*Aluminium-mercury alloys; Electromotive behaviour of —.* R. Kremann and R. Müller. Z. Metallk., 1920, 12, 289–303. Chem. Zentr., 1921, 92, I., 482–483.

ALUMINIUM amalgam was best prepared using a solution of mercuric chloride. The E.M.F. of cells of the type,  $\text{Hg}/0.1N\text{KAl}(\text{SO}_4)_2/\text{Al}_x\text{Hg}$ , was measured. The potential of mercury against the normal calomel electrode was +0.2, that of aluminium -0.82 (constant values could not be obtained on account of the formation of surface layers), and that of the amalgam -1.314 to -1.332 volt, almost independent of the composition. The results indicate the formation of a compound of mercury and aluminium, the composition of which could not be determined. Similarly, in combinations of the type  $\text{Hg}/0.1N\text{NaI}$  in pyridine  $/\text{Al}_x\text{Hg}$ , the potential of the amalgam is, independent of its composition, about 0.25 voltless "noble" than that of aluminium.—E. H. R.

*Magnesium-mercury alloys; Electromotive behaviour of —.* R. Kremann and R. Müller. Z. Metallk., 1920, 12, 303–312. Chem. Zentr., 1921, 92, I., 483.

THE potential of pure magnesium, as determined from the combination,  $\text{Ph}/0.5$  to  $1.0N\text{MgSO}_4/\text{Mg}$  is -1.48 to -1.68 volts. Slight amalgamation brings the value to -1.71 to -1.79 volts. Magnesium and mercury readily combine at the boiling point of the latter. Magnesium amalgams when exposed to the air quickly become covered with brownish-black skin of  $\text{MgO}$ ,  $\text{Mg}(\text{OH})_2$ , and  $\text{MgCO}_3$ , coloured with finely divided mercury; oxidation also occurs in a desiccator. Traces of magnesium bring the potential of mercury to a value 0.3 volt less "noble" than that of magnesium; this value persists up to 85 atoms % Mg, then rises a further 0.17 volt to a maximum at 91 atoms % Mg, afterwards falling again. The maximum may be due to a compound,  $\text{Mg}_2\text{Hg}$ , less "noble" than either component, which is solid at ordinary temperature and forms mixed crystals with magnesium and probably with mercury.—E. H. R.

*Alloys of magnesium with lead and cadmium; Electromotive behaviour of —.* R. Kremann and J. Gmachi-Pammer. Z. Metallk., 1920, 12, 403–406. Chem. Zentr., 1921, 92, I., 484.

EXPERIMENTS on the E.M.F. of the combination  $\text{Pb}/0.5$  molar  $\text{MgSO}_4/\text{Pb}_x\text{Mg}_{1-x}$  indicate the existence of a compound  $\text{PbMg}$ . Alloys with 0–33 atoms % Pb have practically the potential of pure magnesium, those with 33 atoms % Pb up to nearly pure lead have a potential only 0.14 volt "nobler". The heat of formation of the compound is therefore relatively small. The compound rapidly decomposes in air to a black metallic powder consisting of black magnesium suboxide, lead, and, probably lead suboxide. Similar experiments with magnesium-cadmium alloys agree with the equilibrium diagram for these alloys, indicating the existence of a compound  $\text{MgCd}$  0.10 volt "nobler" than

magnesium, 0.86 volt less "noble" than cadmium. This compound also disintegrates quickly in air.  
—E. H. R.

*Metallurgical furnaces; Basis of the heat losses of* —. P. Rosin. Metall. u. Erz, 1920, 17, 463—475; 1921, 18, 37—45, 78—88, 99—104.

A THEORETICAL and mathematical investigation into the factors governing the loss of heat by radiation and convection from furnaces. The four factors governing the magnitude of the losses are the shape of the furnace, the nature of the materials used in its construction, the difference in temperature between the inside and outside surface of the walls, and the time consumed by a unit charge in passing through the furnace. For every furnace on steady work there is a lower limit to the amount of heat lost by conduction, and this amount cannot be reduced by thickening the walls. If the charge completely fills the furnace, less heat proportionately is lost than if it only partly does so, while a large furnace loses relatively less heat than a smaller one. The ideal form for a cylindrical furnace is one in which the height is equal to the diameter; for a rectangular furnace, a cube; and for a prismatic furnace, one having its height equal to the cross-section. A method of determining the heat conductivity of furnace bricks is described, and a table is given showing the results obtained at various temperatures for the principal varieties of firebrick.—A. R. P.

#### PATENTS.

*Steel; High-speed* —. C. M. Johnson. U.S.P. 1,370,020, 1.3.21. Appl., 12.12.19.

A STEEL containing C 0.20—0.75%, Si 0.35—0.80%, Mn 0.35—0.55%, W 12—18%, Cr 3—5%, V 1.1—1.5%, Mo 0.25—1.0%.—J. W. D.

*Iron; Method of making wrought* —. J. Aston, Assr. to A. M. Byers Co. U.S.P. 1,370,622, 8.3.21. Appl., 14.11.19. Renewed 30.6.20.

MOLTEN metal from a steel-making process is granulated (e.g., by dropping vertically) and caused to fall into a bath of iron silicate slag in a container of such shape that a coherent ball or sponge of mixed metal and slag is formed below the level of the slag bath.—C. A. K.

*Iron and steel; Method of and apparatus for the production of* —. E. Riveroll, Assr. to California Electric Steel Co. U.S.P. 1,370,915, 8.3.21. Appl., 12.5.19.

IRON ore is heated by means of reducing and carburising gas under such conditions as to temperature and time that part of the ore is reduced to carburised sponge iron and the other part is unchanged; the reaction is completed by heating the mixture thus formed to complete the reduction and melt the reduced iron.—B. V. S.

*Ferro-chromium; Producing low-carbon* —. F. Krupp A.-G. E.P. 145,709, 26.6.20. Conv., 27.7.16.

HIGH-CARBON ferro-chromium is introduced in molten form into a converter and blown for a longer or shorter period according to the carbon content desired.—J. W. D.

*Ferro-chrome alloys; Production of* —. W. B. Ballantine. E.P. 159,568, 24.11 and 13.12.19, and 13.1.20.

A CHROMIUM compound, e.g., chromite, is reduced by means of a cheap reducing agent, such as calcium hydride, and the resulting crude molten alloy submitted to the action of an oxidising blast in a converter or like converter. The chromium content of the refined alloy is adjusted, before or after tapping from the converter, by means of a thermo-

reducing mixture containing a chromium compound.—J. W. D.

*Electrodes; Arc-welding* —. Wilson Welder and Metals Co., Inc., Asses. of J. Churchward. E.P. 148,265, 16.12.19. Conv., 21.7.19.

A FILM of rust is formed on the cleaned electrode rod by immersion in acidulated water, and the rod is then dipped in milk of lime and dried, the dipping process being repeated until a deposit of sufficient thickness is obtained. After baking at 300° F. (150° C.) the rod may be drawn through a wire-drawing die to the required size. The lime coating becomes incorporated with the porous oxidised surface of the metal, and is not detached by subsequent abrasion or bending of the electrode.

—C. A. K.

*Electrodes for electric arc welding*. H. Ogden, and The British Arc Welding Co., Ltd. E.P. 159,742, 24.1.20.

A BARE electrode is coated uniformly with a flux paste containing not more than 5% of glue or casein by inserting it through an adjustable hole in the end of a rectangular vessel containing the flux mixture. The paste is of such consistency as not to extrude through the hole in the containing vessel.—C. A. K.

*Furnace for subjecting ores or other materials to the action of gases, or the like*. British Oxygen Co., Ltd., S. W. Bray, and I. H. Balfour. E.P. 159,530, 9.10.19.

AN inclined retort extends through a furnace provided with gas burners or other means for heating the retort. The material to be treated is fed into the upper end of the retort in definite quantities from a hopper by means of a sliding or plunger valve. A similar device allows the discharge of the heated material into a closed receiver. Scoops fixed to a rotating longitudinal shaft agitate the material in the retort, and the required atmosphere is obtained by passing a suitable gas into the lower end of the retort and providing an outlet at the upper end.—C. A. K.

*Copper alloys*. Sir W. G. Armstrong, Whitworth, and Co., Ltd., C. J. G. MacGuckin, and O. Smalley. E.P. 159,537, 24.10.19.

ALLOYS containing Cu 80—91%, Al 7—10%, Mn 1—6%, Fe 1—4%, retain their strength up to 450° C., and are especially suitable for the manufacture of turbine blades.—J. W. D.

*Tin; Purification of — or production of tin salts from crude tin*. J. J. Collins. E.P. 159,659, 10.12.19.

IMPURE or crude tin, in a more or less finely divided state, is subjected to the action of stannic chloride, whereby the tin is dissolved and stannous chloride produced, and chlorine gas is injected into the latter so that it continuously absorbs chlorine and gives it up to the metallic tin for the production of further quantities of stannous chloride.

—J. W. D.

*Solder*. A. H. Phillips and C. G. Arnold. E.P. 159,797, 13.7.20.

A SOLDER (m.p. 257° C.), especially suitable for use with cast iron, has the composition 2.2% Co, 10.0% Cu, 0.6% Ag, 0.6% Sb, 86.6% Sn. The cobalt is melted first, and the other metals are added in the above order.—C. A. K.

*Tungsten [silicon] alloy*. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 160,373, 17.4.20.

TUNGSTIC acid, containing a small quantity of silicic acid, is mixed with sufficient magnesium to

reduce the silica, and the mixture is heated in hydrogen. Alternatively the mixture of tungstic acid and silica may be reduced in hydrogen, and the resulting X-metal (cf. E.P. 155,851; J., 1921, 265 A) mixed with the requisite quantity of magnesium powder to reduce the silica and again heated. In either case the magnesium oxide is extracted from the metal powder by means of hydrochloric acid. The resulting metal, containing 1–3% Si, when worked into wire gives a filament that does not sag and shows great resistance to shock.—A. R. P.

**Tungsten; Extraction and recovery of** —. A. G. de Golyer and A. E. Lund. U.S.P. 1,335,277, 30.3.20. Appl., 11.5.16.

THE powdered ore is heated and agitated with an alkali hydroxide or carbonate solution, and a salt of calcium, barium, mercury, lead, or silver is added to the filtered alkali tungstate solution to precipitate an insoluble tungstate.

**Aluminium; Process for making powdered or granulated** —. L. B. Tehbets. U.S.P. 1,327,743, 13.1.20. Appl., 4.12.17.

ALUMINIUM is heated above its melting point and then agitated while it cools. The metal is very brittle at temperatures just below the m.p., and can be readily disintegrated.

**Manganese-silver ores; Treating** —. L. W. Austin. U.S.P. 1,327,974, 13.1.20. Appl., 14.4.19.

REFRACTORY manganese-silver ores are rendered much more amenable to cyanide treatment if they are mixed with pyrites and sulphuric acid, and the product washed with water.

**Ore concentration.** B. H. Dosenbach, Assr. to E. M. Dosenbach. U.S.P. 1,350,364, 24.8.20. Appl., 16.11.17.

A MODIFICATION of the flotation process in which the ore pulp is treated with a gaseous modifying agent such as coal gas or other gas obtained by distillation which has been previously treated with water to remove soluble matter and which effects flotation of certain constituents of the ore.—A. R. P.

**Flotation of minerals.** (A) C. L. Perkins, (b) (c) R. E. Sayre, Assrs. to Metals Recovery Co. U.S.P. (A) 1,370,357, (b) 1,370,366, and (c) 1,370,367, 1.3.21. Appl.; (A) 13.7.20, (b) (c) 2.7.20.

AFTER an addition to the mineral pulp of a small quantity of (a) terpene hydrate, (a) a dihydric alcohol, or (c) a thio-aldehyde compound, a flotation operation is carried out.—C. A. K.

**Flotation of minerals.** R. E. Sayre, Assr. to Metals Recovery Co. U.S.P. 1,370,843, 8.3.21. Appl., 2.7.20.

A SMALL quantity of a ketone condensation product is added to mineral pulp to assist in the mineral concentration during a subsequent flotation operation.—C. A. K.

**Precious metals; Process for extracting** —. C. P. Priest. U.S.P. 1,352,832, 14.9.20. Appl., 21.8.19.

ORES or sands containing precious metals are ground in a solution of sodium chloride, then mixed with a solution of a mercury compound (e.g., mercurio chloride), and the resulting slime electrolysed to produce hydrogen and effect amalgamation of the metal.

**Alloy.** T. Kosugi. U.S.P. 1,369,818, 1.3.21. Appl., 30.6.20.

AN alloy containing Cu 75–90%, Al 7–12%, Ni 2–7%, and Fe 1–6%.—J. W. D.

**[Alloy for] production of gears.** R. D. Babson, Assr. to Baush Machine Tool Co. U.S.P. 1,371,214, 15.3.21. Appl., 3.3.20.

AN alloy containing aluminium about 94%, magnesium about 0.5%, manganese, and copper is subjected to excessive working, then heated to about 500°–525° C., and quenched.—W. J. W.

**Metallic oxides; Reduction of** —. C. M. Johnson. U.S.P. 1,370,021, 1.3.21. Appl., 9.6.20.

THE reduction of the oxide is effected by smelting in the presence of both ferrosilicon and ferrochromium.—J. W. D.

**Leach liquor; Treatment of** —. C. R. Hayward, H. M. Schleicher, and F. O. Stillman, Assrs. to Moa Iron and Development Corp. U.S.P. 1,370,646, 8.3.21. Appl., 8.12.19.

A SOLUTION containing the sulphates of metals of different valencies is first converted into a chloride solution, and the metals are then selectively precipitated by suitable reagents.—A. R. P.

**Metal surfaces; Ornamenting and protecting** —. O. Hommel. U.S.P. 1,370,967, 8.3.21. Appl., 16.5.18. Renewed 5.8.20.

A SOLUTION of a metallic salt is applied to the surface of a metallic article containing aluminium so as to produce an even coating of the salt thereon. The article is then heated to such a temperature that the aluminium reduces the metallic salt with the formation of a coating of the metal which adheres to the underlying metal.—A. R. P.

**Nickel-plating.** T. A. Edison. U.S.P. 1,371,414, 15.3.21. Appl., 17.6.19.

A NICKEL SULPHATE solution is used as an electroplating bath, and portions of it are continuously withdrawn, treated with nickel hydroxide, and returned to the bath.—A. R. P.

**Lead-coating process.** R. J. Shoemaker, Assr. to Leadizing Co. U.S.P. 1,371,445, 15.3.21. Appl., 11.12.20.

A PERMANENTLY adherent coating of lead is formed on iron or steel articles by immersing the metal in a solution containing not less than 35% of lead acetate.—C. A. K.

**Briquetting [metal fragments].** O. C. Duryea and M. C. White, Assrs. to American Briquet Machine Co. U.S.P. 1,371,671, 15.3.21. Appl., 14.2.17.

SMALL metallic fragments are pressed into an approximately solid mass by applying a comparatively mild pressure. A high pressure is then exerted by explosive force, whereby the metal becomes practically welded together.—C. A. K.

**Substances sensitive to oxidation; Process for heating or firing** — e.g., iron, steel, porcelain, and the like. H. Koppers. G.P. 331,924, 14.5.18.

THE articles are heated by means of gas, the quantities of gas and air used being so regulated that in the immediate neighbourhood of the articles a reducing atmosphere free from carbon dioxide is maintained. Iron articles are given a protective coat of clay and graphite or other carbonaceous material that will not carburise the iron, while porcelain articles that are burnt in saggars are covered with a quantity of specially-prepared wood charcoal. This method prevents the scaling of iron or the discoloration of porcelain during heating.—A. R. P.

**Iron and phosphorus; Recovery of** — from slags from iron-smelting. Eisen- und Stahlwerk Hoersch A.-G. G.P. 332,118, 24.6.19.

THE molten slag is treated with chlorine or with

chlorine and carbon monoxide together or successively, and the sublimed iron chlorides are collected in a cooled receiver. The residue may be used as a fertiliser, or worked up for the recovery of its phosphorus content by mixing it with carbon and heating the mixture to 700°–800° C. while passing chlorine or carbon monoxide and chlorine through it, whereby chlorides of phosphorus are evolved and condensed in suitable receivers. The original slag may, alternatively, be mixed with carbon and treated with chlorine when, according to the conditions under which the treatment is carried out, iron or phosphorus chlorides are evolved.—A. R. P.

**Aluminium; Process for deoxidising and refining —.** *Process for deoxidising and refining copper.* A. Strasser. E.P. 141,324 and 142,441, 24.12.19. Conv., 9.4 and 26.4.19.

SEE U.S.P. 1,348,457–8 of 1920; J., 1920, 631 A.

**Alloys.** F. Milliken. E.P. 160,708, 6.7.20.

SEE U.S.P. 1,354,988 of 1920; J., 1920, 754 A.

**Ores and other materials; Method of and means for treating finely-crushed —.** E. T. Middlemiss. U.S.P. 1,370,898, 8.3.21. Appl., 20.5.20.

SEE E.P. 153,320 of 1919; J., 1921, 223 A.

**Electro-deposition; Manufacture of plates or sheets by —.** S. O. Cowper-Coles. E.P. 160,554, 22.12.19.

## XI.—ELECTRO-CHEMISTRY.

**Iron.** Meyer and Zappner. See VII.

**Heating coils.** Sligh, jun. See XXIII.

**Separating mercury and copper.** Böttger. See XXIII.

### PATENTS.

**Electrolysers.** R. Pechkranz. E.P. 146,184, 25.6.20. Conv., 27.6.19.

In an electrolyser in which the surface of the electrolyte is open to the atmosphere, parasitic electrolysis on the edges of the electrodes is prevented by employing vertical electrodes alternating with vertical diaphragms and united integrally therewith. The electrodes are mounted in a grooved frame, but do not extend to the lateral and lower outer edges of the frames, which are immersed in the electrolyte. The block of electrodes carried by the frames is surmounted by a multicellular cover made of insulating material and serving as a separating chamber for emulsions and as a collecting chamber for the gases evolved.—J. S. G. T.

**Electrolytic cell.** L. W. Chuhh, Assr. to Westinghouse Electric and Manuf. Co. U.S.P. 1,348,207, 3.8.20. Appl., 4.4.17.

The cell comprises a container of "film-forming material" (e.g., aluminium, tantalum, magnesium) and a number of electrodes of the same material arranged in an electrolyte, alternate electrodes being connected with terminals of opposite polarity. Each outer electrode has a superficial area about one-half that of an intermediate electrode.

**Heat-proof and insulating materials.** F. J. Harden. E.P. 159,956, 4.12.19.

An acid-resisting, insulating material is made by mixing together about 10 pts. by weight of asphaltum and 5 pts. of coal tar pitch, grinding the cold mixture and mixing therewith 2 pts. of flowers of sulphur, 4 pts. of pumice or other inert powder, 1 pt. of silicate of iron or other metal, 3 pts. of asphaltum or finely divided carbon, and 1.5 pts. of paraffin wax. The whole is damped with petroleum

and heated for about  $\frac{1}{2}$  hr. to a temperature not exceeding the melting point of sulphur. The material may be employed in the manufacture of boxes or containers for electric batteries.

—J. S. G. T.

**Fireproof insulating masses from silicates and combustible substances; Preparation of —.** L. Kern. G.P. 331,026, 30.10.19.

MINERAL acids or their salts, excepting aluminium salts or alums, are added before or after mixing the combustible substance with silicates, and the mixture is thereafter formed, dried, and burnt in known manner. The mineral acids or their salts are dissolved in water and added, in quantities of 0.1–0.5%, to the coal or to the mixture of coal, carbonaceous matter, and silicates.—A. de W.

**Electric furnaces.** E. C. R. Marks. From Scovill Manufacturing Co. E.P. 160,082, 15.3.20.

In a furnace working on polyphase current delivered to the charge by electrodes disposed so as to produce an inner rotating magnetic field, and a weaker outer magnetic field between the electrodes and the furnace wall, an additional magnetic field rotating in planes parallel to the surface of the molten charge is produced by connecting the electrode leads with bus-bars carried down the sides of the furnace and supported either inside or outside the furnace casing. The effect of this field is increased by employing a distributed current carrier comprising a number of bus-bars, which may extend underneath the charge. The additional field, according to its direction, co-operates with either the inner or outer fields, whereby the efficiency of heating and mixing of the charge is increased.—J. S. G. T.

**Electric furnace for calcining magnesia and for similar purposes.** F. S. Newall. E.P. 160,231, 9.12.19.

WITHIN a vertical cylindrical retort is a central stationary cylindrical resistance element, forming an annular chamber through which the material to be calcined travels in direct contact with the resistance element. The retort is supported by a carrier mounted on rollers, and means are provided for imparting a rotary or vibrating motion to it. An internal screw or the like within the retort retards the fall of the material to be calcined and ensures thorough agitation.—J. S. G. T.

**Electric furnaces; Electric system for —.** H. A. Greaves and H. Etchells. U.S.P. 1,370,016, 1.3.21. Appl., 12.9.18.

In an electric furnace with three, or a multiple of three, upper electrodes, and a hearth forming an electric resistance, combined with the "inverted star" system of three-phase transformer grouping, the insertion of a fourth transformer between the common terminal junction of the secondary windings of the original three transformers and the hearth electrode connexion, provides a means of varying the amount of electrical energy used for heating the hearth.—W. J. W.

**Electric furnace.** W. R. Clark, Assr. to Bridgeport Brass Co. U.S.P. 1,370,632, 8.3.21. Appl., 8.9.19.

A COMBUSTION chamber is arranged in the upper part of an electric furnace of the induction type provided with a secondary circuit below the upper surface of the charge, in order to prevent chilling or incrustation of that surface.—J. S. G. T.

**Storage batteries; Filling for [dry] —.** C. A. Allison. From S. Narabayashi. E.P. 160,817, 8.1.20.

A FILLING for dry storage batteries consists of 70% of pumice-stone powder freed from metallic com-

pounds and 30% of shredded asbestos or nitrated cotton. The filling is packed between the battery plates and soaked with a solution of sulphuric acid.—J. S. G. T.

*Storage battery; Electric* — C. J. V. Féry. U.S.P. 1,371,746, 15.3.21. Appl., 24.3.19.

A LEAD negative electrode is arranged horizontally at the bottom of a lead jar containing a "fixed" solution of sulphuric acid, and a positive electrode of lead peroxide is arranged vertically above the negative electrode. The horizontal cross-sectional area of the positive electrode is such that, during the charging operation, an upward movement of  $\text{SO}_4$  ions occurs throughout the height of the jar, whereby the concentration of the acid is maintained constant, and the gases evolved at the negative electrode may escape without reacting with the positive electrode.—J. S. G. T.

*Storage-battery separator.* G. Steerup, Assr. to U.S. Light and Heat Corp. U.S.P. 1,370,064, 1.3.21. Appl., 7.6.19.

Wood is treated so as to increase its porosity, and is then impregnated with ammonia gas. The free ammonia is volatilised and the wood treated with steam at such a temperature and for such a time as would injure the cellular structure of untreated wood.—J. S. G. T.

*Pasting; Two-solution method of* — [Manufacture of electrolyte for dry cells.] W. B. Schulte, Assr. to Burgess Battery Co. U.S.P. 1,370,056, 1.3.21. Appl., 10.6.20.

A PASTE for use as electrolyte in a dry cell is prepared by adding a solution of zinc chloride and ammonium chloride in water to a suspension of starch in a solution of zinc and ammonium chlorides, whereby gelatinisation of the electrolyte is effected.—J. S. G. T.

*Electrolytic gas-generator.* I. H. Levin, Assr. to Electrolytic Oxy-Hydrogen Laboratories, Inc. U.S.P. 1,372,442, 22.3.21. Appl., 22.5.17.

SEE E.P. 127,937 of 1918; J., 1919, 587 A.

See also pages (a) 288, *Electrical separation of dust from gases* (G.P. 331,143). 299, *Nitric acid* (U.S.P. 1,371,789). 300, *Nitrous gases* (G.P. 331,040). 302, *Colloidal metallic oxides* (G.P. 332,200). 303, *Insulating products* (E.P. 142,512). 307, *Electrodes for welding* (E.P. 148,265 and 159,742). 319, *Oxidising nitrogen* (E.P. 159,709). *Water-treating apparatus* (U.S.P. 1,371,814). 322, *Immunising serum* (E.P. 104,688). 323, *Electrolytic treatment of organic substances* (G.P. 310,023).

## XII.—FATS; OILS; WAXES.

*Tallows used for launching ships.* J. J. King-Salter. Inst. Naval Arch., 18.3.21. Engineering, 1921, 111, 405–407.

EXPERIMENTS carried out on five samples of tallow showed that the gradients of the temperature-hardness and temperature-compression curves were proportional to the titre tests (solidif. pt. of insoluble fatty acids). The samples examined had the following titre values: mutton tallow with 15% of stearine,  $48.6^\circ\text{C}$ .; mutton tallow,  $48.1^\circ\text{C}$ .; beef tallow,  $46.1^\circ\text{C}$ .; beef tallow and "C" grade beef tallow,  $48.25^\circ\text{C}$ .; "C" grade beef tallow,  $42.2^\circ\text{C}$ . Hardness tests were carried out in a modified Brinell tester, in which a steel ball  $\frac{1}{8}$ " in diam. was crushed into the surface of the tallow by the impact from a weight of 0.25 lb. falling a distance of 6" on to a steel rod or piston in contact with the upper surface of the ball. Depth of sinkage was registered directly by a lever acting from a collar on the piston to a scale reading to 0.001" by means of a vernier. Both hardness (expressed in arbitrary units) and

compression (in tons per sq. ft.) were found to be proportional at a number of temperatures below the titre figure to the titre number, and the hardness test is recommended for ascertaining the suitability of tallows for launching purposes. The load-sustaining power and also the titre value of an unknown tallow can be conveniently determined by interpolation from reference to curves correlating these figures on known samples. The load-sustaining power at different temperatures may similarly be interpolated. Purified recovered tallow is unsatisfactory owing to its brittle and crumbly character. As a lubricant for use between the general ways and the sliding way, a mixture of "Vaxel" (a lime-resin-oil soap softened with mineral oil) and soft soap proved the most efficacious in inhibiting amalgamation of the tallow surfaces. The effect of boiling a natural tallow for 4 hrs. was to increase the hardness, the hardness factor falling from 101 units to 71 units (a hardness factor of zero on the scale corresponding to an actual hardness of infinity).—A. de W.

*Oxidation of paraffin wax.* (1) Fischer. (2) Fischer and Schneider. (3) Schneider. (4) Schneider and Jantsch. See IIA.

*Montan wax.* Fischer and Schneider. See IIA.

### PATENTS.

*Fatty acids in fats and oils; Neutralisation of* — E. R. Bolton and E. J. Lush. E.P. 159,581, 28.11.19.

THE free fatty acids in an oil or fat are neutralised by subjecting the oil or fat in an inert gas to the action of the chemically equivalent proportion of mono- and di-glycerides of the fatty acids separately prepared, at a temperature of, e.g.,  $250^\circ\text{C}$ . for about 3 hrs. The neutralised product is subsequently heated *in vacuo* in the same reaction vessel to evaporate the water chemically formed. The neutralised oils can be refined by ordinary processes, e.g., for the production of edible oils. —A. de W.

*Fat; Manufacture of* — from spent sulphite cellulose lyes. W. Jeroch, and Reichsausschuss für pflanzl. u. tier. Oele und Fette Ges. E.P. 146,430, 3.7.20. Conv., 24.1.17.

SEE G.P. 305,091 of 1917; J., 1920, 663 A.

*Fat and glue-water.* E.P. 137,842. See XV.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Resins used for making varnishes; Characters of* — P. Nicolardot and C. Cofignier. Chim. et Ind., 1921, 5, 150–156. (Cf. J., 1911, 815.)

AN apparatus for determining the relative hardness of different resins consists essentially of a support for a test-block of the resin and a vertically weighted rod carrying at its lower end a steel ball which presses on the surface of the resin. Usually the ball has a diameter of 2 mm. and the rod is weighted with 5 kg.; the diameter of the impression made in the resin is measured after 5 secs. and again after 10 mins., or at other periods during which the pressure is applied. The test is applied at  $0^\circ\text{C}$ . or at  $25^\circ\text{C}$ . Resins are classified as follows: — *Hard resins:* Zanzibar, Madagascar, Demerara. *Semi-hard resins:* Congo, Benguela, Cameroon, Angola red, Kissel, Brazil, Angola white, Sierra Leone. *Soft resins:* Aucoumea, Klaineana, Hopea odorata, H. dealbata. Various Yellow amber, cloudy amher, kauri, Manila, pontianac.—W. P. S.

*Gum-oleoresin of Boswellia serrata; Extraction of turpentine, resin, and gum from the — without the use of solvents.* G. J. Fowler and M. A. Malandkar. *J. Ind. Inst. Sci.*, 1921, 4, 27–42.

THE turpentine is first removed from the oleoresin by distillation with wet steam. The yield amounted to 8–9%, and the oil, which was pale yellow, had the following constants: sp. gr. 0.8435 at 30° C.,  $[\alpha]_D^{20} = +4.54'$ , fraction distilling below 160° C. 81%, 160°–180° C. 14.4%. The gum-resin remaining after the removal of the turpentine is heated with about three times its weight of water in an autoclave at 30 lb. pressure, whereby the hydrated resin rises to the surface and solidifies on cooling, whilst the gum forms a flocculent precipitate or mucilage on the bottom of the vessel. The resin after dehydration by heating to about 130° C. forms a brown transparent mass, amounting to 54% of the original gum-resin and not differing appreciably in appearance from that extracted by a solvent. It has the following constants: acid value 51.6, saponif. value 113, iodine value 108.1. The gum on drying forms a dark grey mass, amounting to 23% of the original gum resin. It forms a mucilage with water of but feeble adhesive power.—G. F. M.

*Isophony; Constitution of the resin acids of —.* A. Grün. *Z. Deuts. Oel- u. Fettind.*, 1921, 41, 49–52. *Chem. Zentr.*, 1921, 92, I, 496.

FROM a consideration of earlier experiments (*cf.* 1919, 506 A), the author considers that abietic acid does not contain two double bonds, but, like pinene, one double bond and a bridge linkage. A formula is proposed which is claimed to explain better than those hitherto suggested the known properties of the compound and to be in conformity with its origin from pinene. The formation of abietic acid takes place by union of one molecule each of  $\alpha$ - and  $\beta$ -pinene, and subsequent oxidation of a methyl to a carboxyl group. By different modes of combination of the two molecules of pinene, a series of abietic and pimic acids could be formed and others could be formed in an analogous manner from camphene or fenchene.—A. de W.

*Low-temperature tar.* Fischer and Ehrhardt. *See* III.

#### PATENTS.

*Concrete paint or composition impervious to oil, spirit, water, and the like. Anti-corrosive paint or composition.* C. H. Iverson and G. S. Roberts. *E.P.* (a) 159,542, and (b) 159,543, 28.10.19.

A COMPOSITION for coating concrete, etc., consists of the double chlorides of zinc and/or magnesium and ammonium, together with oxides of magnesium and/or zinc, and finely-divided iron, with or without the addition of fillers or extenders, *e.g.*, to every 100 pts. by weight of a mixture of 25 pts. of zinc oxide, 1 pt. of silica, 0.5 pt. of sodium baborate, and 75 pt. of powdered glass, are added 14 pts. of zinc chloride and 1 pt. of ammonium chloride. About 1 pt. of water may be added to 2 pts. of the compound powders, or the water may be added in the form of a saturated solution of the mixed chlorides. A protective paint for zinc, concrete, wood, and so like, consists of a mixture of asphaltum and a volatile solvent with acid-resisting or anti-corrosive substances, *e.g.*, aluminium, calcium, or magnesium stearates or stearates, gum elemi, masticated rubber, gutta-percha, or paraffin wax, and fillers, *e.g.*, asbestos, silica, etc., together with lime-proof pigments ground in a suitable medium, *e.g.*, ferric oxide, lithopone, or zinc oxide ground in linseed oil, tung oil, thinners, or a drying oil varnish.—A. de W.

*Paint or varnish and method of making the same.* M. Darrin, Assr. to The Koppers Co. U.S.P. 1,370,195, 1.3.21. Appl., 10.5.19.

A PAINT or varnish comprises a resin obtained from solvent naphtha subjected to an autoclave process, and linseed oil.—A. de W.

*Paint; Water-resistant —.* R. Plönais. G.P. 301,783, 17.9.16.

THE paint consists of calcium carbonate as a pigment and a mixture of potassium silicate (water-glass) and potassium hydroxide as a medium.—A. R. P.

*Gum from grass trees (Xanthorrhoea); Process for the extraction of —.* H. J. Pooley and J. L. Strevens. *E.P.* 160,080, 12.3.20.

THE outside portions of the grass tree are extracted, at a temperature of not less than 160° F. (71° C.), with the alcoholic solvent derived from the fermentation and rectification of the sweet wort obtained by exhaustion of the inner core of the tree with warm water, such solvent being concentrated to a strength of at least 70% of alcohol. Extraction of the gum may be carried out either by percolation or exhaustion with the vapour of the alcoholic solvent, and the dissolved gum may be separated from the solvent by distillation.—A. de W.

*[Rosin soap] emulsions; Method of making —.* J. A. De Cew, Assr. to Process Engineers, Inc. U.S.P. 1,370,884, 8.3.21. Appl., 11.11.20.

HOT rosin soap containing free rosin is diluted by agitating 1 pt. of hot size with 7 pts. of hot water, with subsequent addition of cooler water.—L. A. C.

*Linoleum, lincrusta, artificial leather, etc.; Production of —.* G. Ruth and E. Asser. G.P. 328,580, 21.6.19.

A MIXTURE of equal parts of chromium or aluminium naphthenate heated to 250°–260° and of a metal salt of naphthenic acid heated to 160° C. is used. A naphthenate partly decomposed by heating to 200° C. may also be used.—H. J. H.

*Oils, boiled oils, varnishes, oily pastes, and luting compositions; Preparation of —.* Rostschütz-Farbwerke Liebreich, G.m.b.H. G.P. 330,670, 25.12.19.

THE oils etc. are heated to at least 240° C. with metals or oxides of metals (CuO, ZnO) conferring little or no siccativ properties, whereby the compound formed remains unsaponified in the presence of alkaline lyes etc. The oils may also be heated with driers and metallic oxides together.—A. de W.

*Shoe-creams, wax-, metal-, wood- and leather-polishes; Preparation of —.* P. Friesenhahn. G.P. 331,050, 5.7.19.

THE usual solid constituents of such preparations are dissolved or emulsified in cyclohexanols or cyclohexanones or their homologues either alone or together with other solvents. The syrupy nature of the products of the hydrogenation of phenols is favourable to the retardation of the drying of the finished products and facilitates emulsification of the solid constituents.—A. de W.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Polythionic chlorides.* Bruni and Amadori. *See* VII.

#### PATENTS.

*Rubber; Manufacture of —.* S. C. Davidson. *E.P.* 159,602, 1.12.19 and 19.1.20.

FRESH latex is treated with a dilute alkaline solution of phenol (*E.P.* 11,470 of 1912, 13,488 of 1913,



and 22,138 of 1914; J., 1913, 799; 1914, 758; 1915, 1020), which acts as a preservative and permits the postponement of further treatment for any desired period. The latex is subsequently coagulated by the addition of a suitable acid or of a suitable mixture of a salt such as magnesium sulphate with sulphuric or pyroligneous acid (*e.g.*, in the apparatus described in E.P. 22,489 of 1914), the dehydration of the resulting coagulum being facilitated by fine perforations in the manner already described (E.P. 151,344; J., 1920, 757 A). The coagulum is then rolled into sheet or crêpe and, if desired, pressed into block. By this procedure the rubber is brought into a marketable condition in a much shorter time than usual.—D. F. T.

**Rubber mix and process of compounding rubber.**  
R. C. Hartong, Assr. to The Goodyear Tire and Rubber Co. U.S.P. 1,370,965, 8.3.21. Appl., 30.1.19.

RUBBER is well mixed with water containing aluminium hydroxide in suspension and the water is driven off from the mixture, which is then vulcanised in the usual manner.—A. R. P.

**Plastic, rubber-like material from cellulose esters; Manufacture of —.** F. Cloutb, Rheinische Gummiwaarenfabr. m.b.H. G.P. 324,944, 18.12.17. Addn. to 319,723 (J., 1920, 563 A).

OTHER high-boiling oils, such as creosote oil, are used instead of anthracene oil, as described in the chief patent.—L. A. C.

**Rubber-like products; Hindering oxidation in synthetic —.** Badische Anilin- und Soda-Fabr. G.P. 330,741, 27.2.18.

THE addition of 1% of  $\alpha$ - or  $\beta$ -naphthol, resorcinol, or quinol, to the material before vulcanisation yields a product which does not deteriorate during storage.—L. A. C.

**Adhesive solutions; Manufacture of highly — from varieties of artificial rubber which are insoluble or soluble with difficulty in the usual solvents, or give solutions of little adhesive power.** Akkumulatoren-Fabr. A.-G. G.P. 331,031, 6.12.17.

THE rubber is heated in air to at least its melting point, *i.e.*, about 140° C., under a pressure of about 1 atm., and is subsequently cooled, cut up, and dissolved in petroleum spirit.—L. A. C.

**Substance resembling rubber; Manufacture of a —.** F. de la Rosée. G.P. 331,334, 15.2.18.

RESIN, *e.g.*, balsam resin, is melted with calcium chloride, and repeatedly distilled with further additions of calcium chloride and bleaching powder. A mixture of the oily product with rubber or rubber regenerates is then vulcanised.—L. A. C.

**Caoutchouc; Art of vulcanising —.** C. W. Bedford, Assr. to The Goodyear Tire and Rubber Co. U.S.P. 1,371,662—4, 15.3.21. Appl., 6.1.17, 19.3.19, and 17.5.19.

SEE E.P. 130,857 of 1918; J., 1919, 731 A.

## XV.—LEATHER; BONE; HORN; GLUE.

**Depsides and tannins.** K. Freudenberg. Collegium, 1921, 10—19.

A HISTORICAL account of Fischer's synthesis of gallotannic acid and tannin-like substances. Carbo-methoxybenzoic acid was prepared from *p*-hydroxybenzoic acid and was converted, by means of the Schotten-Baumann reaction, into an ester anhydride of *p*-hydroxybenzoic acid. This was termed a depside. By partial saponification of triacetyl-gallic acid to diacetyl-gallic acid and combination of the

free hydroxyl group with triacetyl-galloyl chloride, a penta-acetyl-*m*-digallic acid was obtained, which proved to be a true tannin, like the corresponding didepsides of the dihydroxybenzoic acids. There yet remained the 7% of dextrose obtained from gallotannic acid, which was not easily liberated, and hence was not present in the form of a glucoside. The absence of free carboxyl groups in gallotannic acid indicated that the carboxyl groups in gallic acid were united to the hydroxyl groups in the dextrose. Fischer and Bergmann prepared penta-digalloylglucose and found it very similar to Chinese gallotannic acid. It was not identical, since the natural product consists of a mixture of isomers, whilst the synthetic product was a single substance. The synthesis of fully methylated gallotannic acid was also effected. The comparison of pentagalloylglucose with Turkish gallotannic acid was rendered difficult owing to the presence of an ellagic acid tannin in the natural product. Glucogallin, a constituent of rhubarb, was identified by Fischer and Bergmann as 1-galloyl- $\beta$ -glucose, and was synthesised from acetobromoglucose and silver triacetyl-gallate. Fischer worked out a new method for preparing partially acetylated glucose derivatives. Two molecules of acetone are combined with one of dextrose in presence of a little hydrochloric acid, forming a diacetoneglucose. The free hydroxyl group is combined with an acid chloride and the acetone is split off one molecule at a time. Further developments have been along the lines of analysis with tannase which has helped to explain the constitution of chlorogenic acid ( $C_9H_8O_6$ ). By its aid, hamamel-tannin was shown to consist of a new sugar esterified with two molecules of gallic acid. Chebulinic acid from myrobalans contains a digalloylglucose combined with an unknown acid. There are four groups of tannins, ester tannins, catechins, oak and chestnut, and ellagic acid tannin. Crystalline tannins are difficultly soluble in water, amorphous tannins are more readily soluble.—D. W.

**Oakwood extracts; Cause of the development of gas in —.** F. Liebert. Collegium, 1921, 76—77.

A SAMPLE of strongly fermenting extract was inoculated on a plate of malt extract-gelatin and kept at 22° C. for 2 days. A pure yeast culture was obtained which closely resembled *Saccharomyces apiculatus*. This is an extraordinary instance of selective growth of yeasts to the exclusion of bacteria and moulds. The infection was traced to exposure of the concentrated extract to the air after being run out of the evaporator. Covers and disinfection prevented it. The acidity of the extract also helped the growth of the yeast, and an extract neutralised with calcium carbonate would not ferment.—D. W.

**Tannin analysis. I.** V. Kuhelka and B. Köhler. Collegium, 1921, 77—83.

AFTER shaking solutions of certain solid pine bark and oak wood extracts with hide powder in accordance with the provisions of the official method of analysis, and filtering, the filtrate gave a precipitate with gelatin and with iron salts and was coloured. A fresh portion of hide powder absorbed a large amount of matter from this filtrate and rendered it "tannin-free" according to qualitative tests. Similar observations were made with liquid extracts, but the maximum amount of matter absorbed from the first filtrate (10%) was obtained with solid extracts. Samples of an oak bark extract tested by four different analysts gave results showing variation in tans from 38.6% to 45.7% by the "shake" method. In tanning practice with some materials, the hide is first entered into nearly exhausted liquors, whereas in the analysis the powder is treated with freshly made



liquor. The "shake" method was worked out for certain tanning materials and is not suitable for some new products. The principle of the official method is wrong, since all those substances which are not quickly absorbed are classified as non-tans, although they may be capable of being absorbed by hide powder and react with gelatin and iron salts. In the filter method of analysis, the first layers of hide powder are saturated with tannin from the fresh strong tannin solution and then the other layers come into contact with the partially deannised solution. Where the "shake" method does not give completely detannised solutions, the "filter" method should be employed. Tables are appended showing the suitability of the "shake" method of analysis for quebracho, mimosa, chestnut, and sumach extracts and the efficacy of the double shake method and filter bell method with akwood, oak and pine bark extracts.—D. W.

*Tanning [and dyeing] rabbit skins for furs.* G. Desmurs. *J. Soc. Leather Trades' Chem.*, 1921, 5, 84—87.

**S**KINS of rabbits and similar animals are tanned in liquors made up from a mixture of 100 pts. of unphosphated quebracho tannin (40% tans), 10 pts. of much extract (22% tans),  $\frac{2}{3}$  pt. of anhydrous sodium sulphate, and  $\frac{1}{3}$  pt. of aluminium sulphate. A similar mixture is also suitable for tanning sheepskins in the paddle. After a preliminary treatment with sodium carbonate, ammonia, or caustic soda, the furs are dyed black with logwood and stic and mordanted with copper sulphate or pyrochrome of iron. Grey, brownish-black, or greyish-brown shades can be obtained with *p*-phenyleneamine, *o*- and *p*-aminophenol, *m*-toluylenediamine and hydrogen peroxide.—D. W.

*Formaldehyde tannage.* VI. W. Moeller. *Collegium*, 1921, 20—31. (*Cf. J.*, 1920, 36 A, 730 A.)

**F**ORMALDEHYDE is absorbed in large amounts by animal charcoal under special conditions. The volume and concentration of the formaldehyde solutions and the time factor have been overlooked in previous researches. The assertion that formaldehyde does not act on agar-agar is not borne out by experiment. The action of dilute solutions of formaldehyde on hide powder has been determined. 55% of the formaldehyde present in a 3.5% solution was adsorbed by the hide powder after 14 days' contact. The amount of formaldehyde absorbed increases with the length of time of contact. Similar results were obtained with neutralised solutions of formaldehyde, but much less was absorbed from concentrated solutions. The addition of alkali even to neutralise the formaldehyde solution causes the Cannizzaro inversion to proceed to a certain extent, the polymerisation of formaldehyde, and further the reaction of the decomposition products with a certain portion of the formaldehyde to form methyleneamino-acids. These reactions must cause the neutralised formaldehyde solution to react acid, and three different solutions of formaldehyde were found to be acid when in contact with hide powder for 14 days. Methyleneamino-acids have a hydrolysing action, hence the "proteolytic-constant" of hide powder is increased by formaldehyde solutions. The lack of permanency in formaldehyde-tanned leathers is easily explained by insufficient washing of the tanned leather. Methyleneamino-acids remain in the leather and they slowly hydrolyse the leather, causing brittleness. There is not as much of hide substance with 15—30% solutions of formaldehyde owing to the more intense tannage. The total acid formed in these concentrated solutions is the same as that formed in the dilute solutions. Samples of powder tanned with formaldehyde solutions of different strengths were extracted

with water and those tanned in concentrated solutions were found to be less stable to the solvent action of water, and powders tanned in neutral formaldehyde solutions were less stable than the others. The small amount of formaldehyde-tannin taken up in alkaline solution gives all the desired properties required for aldehyde-leather, and the absolute amount of formaldehyde-tannin taken up is not a vital factor.—D. W.

*Mineral tannages.* IV. W. Moeller. *Collegium*, 1921, 67—75.

**N**EUTRALISED and unneutralised solutions of ferric chloride of different strengths (1.1%—10.9%  $\text{Fe}_2\text{O}_3$ ) were allowed to be in contact with hide powder for periods varying from 1 day to 3 months. Half the total iron absorbed was taken up the first day, after which there was very little increase for a month. Neutralisation of iron salts does not produce a fully satisfactory tanning solution. The electrolytic dissociation of iron salts is excessive and results in hydrolytic decomposition of the hide after prolonged contact. As much as 67% of the hide powder was dissolved by the strongest solutions after 3 months' contact. This explains the "loose" fibre and tendency to become brittle of iron-tanned leathers. The ready dissociation of all iron salts even in neutral solution causes the difficulties connected with iron tannages and renders it difficult to obtain a permanently peptised system. The acid peptiser is continuously dissociating and the acid portion effects the hydrolysis of the hide and the decomposition of the leather. Unlike other tannages, there appears to be no limit to the amount of iron which can be absorbed by the hide. The assertion that mineral salts such as chromium and ferric salts can tan without the addition of soda is not borne out by experiment. The hydriions in such unneutralised solutions decompose the hide with which they are in contact and the hide decomposition products act just like any other alkali. The basic groups combine with the acids and a more or less peptised system results from the presence of these basic decomposition products. The longer such a solution is in contact with the hide substance the more tanning effect it will exert upon the hide. Such a process of tanning is useless for practical purposes, since it does not give a satisfactory leather. Modern processes for iron tannages provide acceptors for the hydriions in the tanning salts in order to obviate their hydrolytic action during tannage and storage.—D. W.

*Gelatin jellies; Elastic properties of —.* S. E. Sheppard and S. S. Sweet. *J. Amer. Chem. Soc.*, 1921, 43, 539—547.

**M**EASUREMENTS of the modulus of rigidity of gelatin jellies under torsion show that they follow Hooke's law nearly to the breaking point, the elastic limit nearly coinciding with the tenacity (*cf. J.*, 1920, 758 A). The expression  $E = kc^n$  expresses the relation between the modulus of elasticity,  $E$ , and the concentration,  $c$ , but the constants  $k$  and  $n$  differ for different grades of gelatin. The elasticity is not a simple function of hydrogen ion concentration in the case of acid jellies. Alcohol and glycerol increase the jelly strength up to a certain concentration. Jellies with more than 40% of alcohol separate into two phases under torsion. The bearing of the results on theories of jelly structure is discussed. (*Cf. J.C.S.*, May.)—E. H. R.

*Colloidality; The zone of maximum —. Its relation to viscosity in hydrophile colloids, especially karaya gum and gelatin.* J. Alexander. *J. Amer. Chem. Soc.*, 1921, 43, 434—440.

**T**HE viscosity of reversible emulsoid or hydrophile colloids sometimes increases as the dispersed phase becomes finer, as in oil emulsions, and sometimes

as it becomes coarser, as in the case of soaps. There appears, in fact, to be a "zone of maximum colloidality," the size of particles giving maximum viscosity being probably different for different substances. This zone of maximum colloidality is not limited to so-called emulsoid colloids. Thus with steel the  $\text{Fe}_3\text{C}$ -Fe dispersion reaches its maximum hardness in martensite and becomes softer if the dispersion becomes greater (austenite) or less (troostite, sorbite, pearlite). Experiments in which karaya gum of different degrees of fineness was dispersed in water showed that the viscosity increased with increasing fineness of the gum particles from 5- to 10-mesh (about 4 mm.) to 200-mesh (about 0.1 mm.). The hydration and dehydration of karaya gum and gelatin is discussed. The decrease in the viscosity of highly degraded glue or gelatin is probably due to an increase in the dispersion. Both the jelly strength and viscosity of cold aqueous dispersions of commercial ground glues and gelatins increase with decrease in size of particle, but the optimum conditions for maximum viscosity are not identical with those for maximum jelly strength. (Cf. J.C.S., May.)—E. H. R.

*Proteins [gelatin]; Method of purifying certain kinds of —.* A. M. Field. J. Amer. Chem. Soc., 1921, 43, 667—668.

IN consequence of its amphoteric properties (J., 1919, 331 A), any protein with iso-electric point at a hydrogen-ion concentration greater than that of water will lose any combined basic elements when dialysed sufficiently long against dilute acid, and the resulting protein-acid compound can then be completely converted into protein by dialysis against water. Gelatin purified by this procedure forms an opaque white jelly, free from ash. Similarly, proteins with iso-electric points less than that of water in respect of hydrogen ions may be successively treated with dilute alkali and water.

—J. K.

*Proteins [gelatin]; Ion series and the physical properties of —. III. Action of salts in low concentration.* J. Loeb. J. Gen. Physiol., 1921, 3, 391—414.

THE Hofmeister lyotropic series is founded on an error due to a failure to notice the influence of the added salt on  $p_H$ . At the same  $p_H$  the effect of ions on the swelling, osmotic pressure, and viscosity of gelatin depends only on the sign and valency of the ions, and univalent ions of the same sign have practically the same effect. Divalent and still more trivalent ions have a greater effect but do not differ much among themselves if they have the same sign. (Cf. J.C.S., May.)—G. B.

*Proteins [gelatin]; Colloidal behaviour of —.* J. Loeb. J. Gen. Physiol., 1921, 3, 557—564.

THE potential difference between gelatin chloride solutions inside a collodion bag and an aqueous solution outside is depressed by a neutral salt in the same proportion as the osmotic pressure is depressed; the potential difference can be calculated by Nernst's formula if it is assumed to be due to the difference in the  $p_H$  measured inside and outside the bag, which latter difference seems to result from the Donnan membrane equilibrium. (Cf. J.C.S., May.)—G. B.

*Anthrax spores.* Müller. See XIXb.

#### PATENTS.

*Tanning of hides and skins; Apparatus for rapid —.* F. Gilardini. E.P. 160,422, 5.12.19. Addn. to 114,631 (J., 1919, 297 A).

RELATES to mechanical improvements in the apparatus described in the chief patent.—D. W.

*Colouring-matter for leather and method of using the same.* J. H. Pfingsten, Assr. to Presto Color Co. U.S.P. 1,371,572, 15.8.21. Appl., 2.7.17.

PULVERULENT, insoluble pigment is suspended in water, and the leather is agitated therein in the presence of heat, so that the pigment is uniformly deposited in the interstices of the leather.—D. W.

*Glue-water; Apparatus for treating mixtures of fat and — derived from boiling and drying organic substances.* K. Niessen. E.P. 137,842, 13.1.20. Conv., 2.10.16.

TWO receptacles are arranged side by side with their lower parts connected by a siphon for equalising the pressure. One receptacle of such size that it can hold not only the maximum output of fat from the steriliser but also a small quantity of glue water, serves as fat separator and the other as glue-water holder and evaporator. Both receptacles are connected with a steriliser and glue extractor by means of a series of pipes fitted with valves. By operating the valves the glue-water and fat separate in the fat separator, which is emptied by means of another valve, and the glue-water is returned to the extractor.—D. W.

*Ferro-leather; Process for producing —.* W. Mensing. U.S.P. 1,371,803, 15.3.21. Appl., 23.1.17. Renewed 27.1.21.

SEE G.P. 314,885 of 1916; J., 1920, 199 A.

*Artificial leather.* G.P. 328,580. See XIII.

## XVI.—SOILS; FERTILISERS.

*Soils; Rate of formation of soluble substances in several organic —.* M. M. McCool and L. C. Wheeting. Soil Sci., 1921, 11, 233—247.

IN organic soils, for a given moisture content, the rate of formation of soluble material varies with the temperature. At higher temperatures (25° C.) optimum moisture conditions tend to bring greater amounts of material into solution than when the soil is saturated. The reverse is true at lower temperatures (7° C.). In moist soils the concentration of soluble substances generally increases on standing up to a certain point and then decreases. In organic soils the amount of soluble substances present and the rate at which they are produced vary with the depth. In general the ability to yield soluble materials decreases regularly from the surface to the water-table, the muck soils studied being very inactive below a depth of 2 ft. The zone of weathering and the region of greatest activity coincide.—W. G.

*Determination of potash as perchlorate.* Jarrell. See XXIII.

#### PATENTS.

*Fertilising material, and process of producing same.* M. O. Johnson. U.S.P. 1,370,117, 1.3.21. Appl., 15.12.19.

VEGETABLE fibrous and fruity matter and coagulated vegetable proteids (cf. U.S.P. 1,362,868; J., 1921, 127 A) are compressed into a cake, for use as a fertiliser.—W. J. W.

*Fertilisers, especially calcium cyanamide; Prevention of dustiness in —.* W. Schwarzenauer. G.P. 304,965, 28.3.16.

OILS, obtained by distillation of bituminous shale, are absorbed by a suitable material, such as pulverised shale or the residue from the shale distillation, and the mixture is incorporated with the fertiliser.—W. J. W.



latter does not behave as an aldehyde. By the use of alcoholic ammonia in place of aqueous barium hydroxide for the hydrolysis of glucal triacetate the authors have succeeded in preparing crystalline glucal, needles, m.p. about 60° C.,  $[\alpha]_D^{20} = -7.2$  in aqueous solution, which, like rhamnal, does not show aldehydic properties. One of the impurities which confers strong reducing powers on syrupy "haryta" glucal is 2-desoxy-glucose. On oxidation with benzoper-acid glucal is converted into mannose. (Cf. J.C.S., May.)

—H. W.

Karaya gum. Alexander. See XV.

Starches. Arpin. See XIXA.

### XVIII.—FERMENTATION INDUSTRIES.

*Saccharase [invertase] preparations; Preparation of highly active* —. O. Svanberg. Z. physiol. Chem., 1920, 109, 65—98. (Cf. J., 1920, 636 A, 795 A; 1921, 191 A.)

HIGHLY active invertase preparations were made by autolysing yeast, extracting the residue with water, and fractionating by means of alcohol, protein being removed from the resulting enzyme solutions by treatment with kaolin. A purer preparation was obtained by dialysis.

*Yeast; Nutritional requirements of* —. III. *Synthesis of water-soluble B*. V. E. Nelson, E. I. Fulmer, and R. Cessna. J. Biol. Chem., 1921, 46, 77—81.

A YEAST was sub-cultured 180 times on a salt and sucrose medium, and yet contained enough vitamin B to cure rats. The vitamin, therefore, must have been synthesised by the yeast, which result invalidates Williams's method of estimating this vitamin (cf. following abstract). (Cf. J.C.S., May.)

—G. B.

*Vitamins and yeast growth*. R. J. Williams. J. Biol. Chem., 1921, 46, 113—118.

THE author's quantitative test for vitamin B (J., 1919, 692 A; 1920, 608 A) gives results fairly concordant with Osborne and Mendel's rat-feeding experiments, but bakers' yeast, by the yeast method, shows a much higher vitamin content than brewers' yeast, whereas for animal experiments the reverse holds good. "This is interpreted to mean that there is some specificity in growth stimulants, but not necessarily two totally different substances stimulating growth of the two varieties of yeast" (cf. preceding abstract).—G. B.

*Acetone fermentation process in India*. G. J. Fowler, Y. D. Wad, and A. G. Gokhale. J. Ind. Inst. Sci., 1921, 4, 1—15.

As raw material for the acetone fermentation process, mahua flowers (*Bassia latifolia*), containing 40—60% of total sugars, 0.65—1.1% of nitrogen, and little or no starch, were tried under various conditions but gave very unsatisfactory results. Good fermentations were obtained, however, with starch-containing materials, such as rice, but jawari (chulam) flour was preferred on account of its cheapness. In small scale experiments 1200 g. of jawari flour containing about 52% of starch and 1.2% nitrogen gave about 70 c.c. of acetone and 128 c.c. of butyl alcohol. The most vigorous fermentation took place with cultures only two or three generations from spores, and it is important to ensure that the cultures do not lose their power to sporulate, as they tend to do after continued sub-culture in simple maize mash. This was achieved by inoculating in a mash containing meat extract, peptone, or gelatin. The chief difficulty in large

scale work is the maintenance of sterility up to the point of inoculation 4 hrs.' heating under 15 lb. steam pressure was found to be requisite. The problem of the disposal of the effluent, which amounts to 50,000 galls. per ton of acetone produced, was studied. A cake utilisable for cattle food is obtained by pressing the husk along with the sludge resulting from plain sedimentation. The turbid liquid remaining is clarified by treatment with excess of lime, and gives a further sludge which has valuable manurial properties. In connexion with the above work rough experiments were made on the flashing and explosive concentrations of mixtures of acetone vapour and air, indicating that a concentration of acetone vapour up to 2.3% was safe. The mixture flashes above this concentration, reaching a maximum violence at a concentration of 5.61% of acetone, and settling down to a quiet flame at 10.2% of acetone vapour.—G. F. M.

*Iron; Determination of small quantities of* — in organic liquids, especially in wines. P. Malvezin and C. Rivalland. Ann. Chim. Analyt., 1921, 3, 90—92.

THE ash from a portion of the liquid (e.g. from 20 c.c. of wine) is dissolved in 10 c.c. of 5% hydrochloric acid, the solution filtered and treated with 0.5 c.c. of hydrogen peroxide; after 15 mins., the solution is boiled to decompose excess of peroxide, 5 c.c. of 1% copper sulphate solution and 1 c.c. of 2% sodium salicylate solution are added, the mixture is boiled and titrated with thiosulphate solution, the end-point being denoted by the disappearance of the violet coloration (ferric salicylate). The copper sulphate acts as catalyst in the reduction of the ferric salt.—W. F. S.

*Yeast-nucleic acid*. Thannhauser and Sachs. See XX.

*n-Butyl alcohol-water mixtures*. Wad and Gokhale. See XX.

### PATENTS.

*Marine alga; Utilisation of* — for the manufacture of acetic and butyric acids [by fermentation.] L. Dupont, Assr. to Darrasse Frères. U.S.P. 1,371,611, 15.3.21. Appl., 6.3.19.

SEE E.P. 123,325 of 1919; J., 1920, 499 A.

*Reduction of organic compounds*. G.P. 330,812. See XX.

### XIXA.—FOODS.

*Dough; Relation between the mechanical properties of* — and bread-making. M. Chopin. Bull. Soc. d'Encour., 1921, 133, 261—273.

AN apparatus is described and figured by means of which it is possible to measure the tenacity of a dough in terms of the effort necessary to cause a sample to assume a given shape in a constant time, and to measure the capacity of the dough to stretch into a thin membrane. It is shown that temperature, composition of the dough, method of working the dough, age of the flour, and length of time the dough is allowed to stand, all have an influence on the results. Measurements with numerous samples of flour show that the difference between the specific volume of bread which can be obtained with a flour and the initial specific volume of the dough from this flour is proportional to the square root of the coefficient of extension of this dough developed in a thin membrane. Thus it is possible to determine the index of swelling during bread-making and the yield of bread from a given flour.—W. G.

*Starches, flours, etc.; Commercial classification of* —. Arpin. *Ann. Chim. Analyt.*, 1921, 3, 74—84.

It is suggested that the word flour be used to describe ground cereals, that the word starch be reserved for the amylaceous products obtained from cereals and grains, and that the amylaceous products of tubers, roots, etc., be called "fecula." Analyses and microscopical characters of these different classes of material are given.—W. P. S.

*Milk; Titration of organic acids in* —. B. Kramer and C. H. Greene. *Proc. Amer. Soc. Biol. Chem. J. Biol. Chem.*, 1921, 46, xxxviii—xxxix.

THE application of Van Slyke and Palmer's method for the estimation of titratable organic acid in urine (*J. Biol. Chem.*, 1920, 41, 567) to milk is described. 10 c.c. of milk, 50 c.c. of distilled water, 2–5 c.c. of 20%  $\text{CuSO}_4$ , and 10 c.c. of 10% suspension of  $\text{Cu}(\text{OH})_2$ , are successively put into a 100 c.c. volumetric flask, repeatedly shaken, made up to 100 c.c. after 15 min., and filtered through a Buchner funnel. 50 c.c. of the filtrate is titrated with  $N/10$  hydrochloric acid, using first 0.5 c.c. of 1% phenolphthalein as indicator, and when this has been decolorised ( $p_H=8$ ), adding 5 c.c. of 0.02% "tropaeolin 00" and titrating further until the solution matches the standard (0.6 c.c. of 0.2  $N$   $\text{HCl}$ , 5 c.c. of "tropaeolin 00," and water to 60 c.c.). Similar large test tubes are used, and a corrective is applied for the 0.1  $N$  hydrochloric acid used in the blank titration of distilled water from  $p_H 8.0$  to 2.7.—G. B.

*Milk; Dialysis indicator method for the determination of the hydrogen ion concentration of* —. B. Kramer and C. H. Greene. *Proc. Amer. Soc. Biol. Chem. J. Biol. Chem.*, 1921, 46, xlii—xliii.

THE dialysis indicator method of Levy, Rowntree, and Marriott is used. 5 c.c. of milk is dialysed in a collodion sac against an equal volume of 0.9% sodium chloride solution; the level of the salt solution is above that in the sac. After at least 5 mins. the external solution is colorimetrically compared with phosphate or acid phthalate standards and Clark and Lubs' indicators. Parallel electrometric estimations showed excellent agreement. The following values for  $p_H$  were found: Human milk, 6.8; cow's milk, 6.4; protein milk, 4.8; butter-milk, 4.4. (*Cf.* Lovatt Evans, *J. Physiol.*, 1921, 54, 353.)—G. B.

*Goat's milk; Acidity of* — in terms of hydrogen ion concentration, with comparisons to that of cow's and human milk. E. W. Schultz and L. R. Chandler. *J. Biol. Chem.*, 1921, 46, 129—131.

AVERAGE goat's milk has  $p_H 6.53$ , when fresh, and 3.92 when fully soured. For fresh cow's milk Van Slyke and Baker (*J.*, 1920, 130 A) found 6.5–7.2, mostly 6.5–6.76, and other authors 6.52–6.8; the maximum acidity of soured cow's milk is 4.65. Human milk has  $p_H$  between 6.86 and 7.46.—G. B.

*Milk; Iron as the cause of formaldehyde and diphenylamine reactions [for nitrates] of* —. F. Reiss. *Z. Unters. Nahr. Genussm.*, 1921, 41, 26—29.

MILK containing iron yields reactions with formaldehyde and diphenylamine-sulphuric acid reagents indicating the presence of nitrates. The iron may be derived from the cans in which the milk is kept; the colorations obtained can be attributed to the presence of nitrates only when the milk is proved to be free from hydrogen peroxide and from dissolved iron.—W. P. S.

*Milk-powder; Determination of moisture in* —. N. Schoorl and S. C. L. Gerritzen. *Pharm. Weekblad*, 1921, 58, 370—378.

THE determination of moisture in milk-powder by heating for 3–4 hrs., either at 95° or at 103° C., gives results which are 1.6–1.9% too low, as compared with the correct moisture found by exposure *in vacuo* over phosphorus pentoxide for 24 hrs. at 95° C. Addition of sand to the sample does not influence the results. On prolonged heating at 95° C. a very gradual loss in weight takes place, but even after 29 hrs. the correct moisture is not indicated. If drying is continued at 103° C., there is a progressive loss of weight, and at a certain stage decomposition of the milk sugar sets in owing to presence of phosphates in the milk-powder. By drying in air which has been passed through sulphuric acid, a correct moisture figure may be obtained after 36 hrs. A rapid method for moisture determination, giving results correct within 0.1–0.3%, consists in heating the milk-powder in undried air at 110° C. for a period not exceeding 2 hrs., the weighing bottles being placed on an asbestos sheet in the oven to avoid risk of decomposition.—W. J. W.

*Casein solutions; Chemical and physical behaviour of* —. J. Loeb. *J. Gen. Physiol.*, 1921, 3, 557—564.

EXTENSION to casein solutions of conclusions previously arrived at for gelatin and egg albumin that the forces determining the combination between proteins and acids or alkalis are the same forces of primary valency which also determine the reaction between acids and alkalis with crystalloids, and that the valency and not the nature of the ion in combination with a protein determines the effect on the physical properties of the latter. (*Cf.* J.C.S., May.)—G. B.

*Proteins; Digestibility of* — in vitro. I. Effect of cooking on the digestibility of phaseolin. H. C. Waterman and C. O. Johns. *J. Biol. Chem.*, 1921, 46, 9—17.

THE protein of the navy bean is rendered more digestible by cooking; 5 minutes' cooking gave a detectable, and 45 minutes' cooking a maximum increase in digestibility.—G. B.

*Proteins; Physicochemical method of characterising* —. E. J. Cohn. *Proc. Amer. Soc. Biol. Chem. J. Biol. Chem.*, 1921, 46, iii—iv.

THE hydrogen ion concentration at iso-electric point gives the ratio of the acid to the basic dissociation constants (1). By plotting the ratio of acid to protein—and of protein to base (expressed as mols. of  $\text{HCl}$  or  $\text{NaOH}$  added to 1 g. of anhydrous iso-electric protein)—against hydrogen ion concentration, a titration curve is obtained, and a slope of a tangent to this curve at the iso-electric point represents the apparent strength of the multivalent protein as acid or base (2). Values of (1) and (2) together with the hydrogen ion concentration at the iso-electric point are tabulated for five proteins and show considerable differences.—G. B.

*Lecithin. III. Fatty acids of lecithin of the egg-yolk.* P. A. Levene and I. P. Rolf. *J. Biol. Chem.*, 1921, 46, 193—207.

METHODS are given for obtaining lecithin (from egg yolk powder) free from fats, cerebrosides, and saturated lipoids, and with a minimum content of amino nitrogen. On hydrolysis about half the fatty acid was found to be oleic acid, the other half a mixture of palmitic and stearic acids. These facts suggest the existence of more than one lecithin in egg-yolk.—G. B.

*Foods; Determination of alkalinity and phosphates in the ashes of —.* J. Tillmans and A. Bohrmann. *Z. Unters. Nahr. Genussm.*, 1921, 41, 1—17.

A PORTION of the ash is boiled with an excess of  $N/10$  acid, 30 c.c. of 40% calcium chloride solution is added, and the excess of acid is titrated with  $N/10$  alkali, using phenolphthalein as indicator; the number of c.c. (A) of  $N/10$  acid used is a measure of the alkalinity (carbonate plus oxide). The carbonate may be determined separately. Another portion of the ash (at least 0.2 g.) is boiled for 1 hr. with 100 c.c. of  $N/10$  hydrochloric acid, cooled, filtered, and titrated with  $N/10$  sodium hydroxide solution, using methyl orange as indicator; the number of c.c. of  $N/10$  acid neutralised by the ash is noted (B). Twenty c.c. of saturated sodium oxalate solution is added and the titration is continued until the mixture is neutral to phenolphthalein (C). If the ash is alkaline, orthophosphates (Y) only can be present and their quantity, in mg. of  $PO_4$ , is given by the formula  $Y = 3/2(B-A) \times 3.167$ . When the ash is not alkaline, and C has a positive value, orthophosphates and pyrophosphates (Z) are present; in this case  $Y = 3C \times 3.167$  and  $Z = 2(B-2C) \times 4.75$ . If C has a negative value, pyrophosphates and metaphosphates (U) are present; then  $Z = 2B \times 5.75$  and  $U = -C \times 9.5$ . Milk ash contains very little true alkalinity, and about 30—40% of phosphate ions in the form of orthophosphate; pyro and metaphosphates are not present. Flour ash is free from true alkalinity and contains a mixture of phosphates. Meat (beef and horse) ash is also free from oxides and carbonates but contains a mixture of ortho and pyrophosphates (6—7% as ortho and 47—50% as pyrophosphate ions). Fruit juice ashes consist mainly of carbonate and the phosphate is present wholly as orthophosphate. The alkalinity of cocoa ash consists of about one-third oxide and two-thirds carbonate; orthophosphate is present.—W. P. S.

*Pepsin; Use of edestin in determining proteolytic activity of —.* J. F. Brewster. *J. Biol. Chem.*, 1921, 46, 119—127.

A MODIFICATION of the method of Fuld and Levison (*J. Chem. Soc.*, 1907, ii., 76) and a method for preparing pure edestin from hemp seeds are described. After mixing various amounts of 1% edestin and  $N/10$  hydrochloric acid solutions with equal amounts of 10% sodium chloride in a series of tubes, equal amounts of 1% pepsin are added, and the time necessary to produce a clear solution is noted.

—G. B.

*Antiscorbutic properties of commercially dried orange juice.* J. F. McClendon, W. S. Bowers, and J. P. Sedgwick. *Proc. Amer. Soc. Biol. Chem. J. Biol. Chem.*, 1921, 46, ix—xi.

THE dried preparation (*cf.* following abstract) was about as active in preventing guinea-pig scurvy as an equivalent of fresh juice, so that drying did not significantly impair the antiscorbutic properties.

—G. B.

*Fat-soluble vitamin.* H. Steenbock, M. T. Sell, P. M. Nelson, and M. V. Buell. *Proc. Amer. Soc. Biol. Chem. J. Biol. Chem.*, 1921, 46, xxxii—xxxiii.

AS in the case of yellow and white maize, the fat-soluble vitamin content runs parallel to the content of yellow pigment in different varieties of carrots, sweet potatoes, and squash. In yellow maize the vitamin is localised in the endosperm, which, in distinction from the embryo, contains most of the pigment. Carotin after numerous crystallisations, is still active on rats, but, on the other hand, very active cod-liver oil may be free from yellow pig-

ment. Light, which destroys the pigment, also destroys vitamin. The latter, obtained from alfalfa (lucerne) hay, was not destroyed by hot saponification, nor by hydrogen peroxide or nascent hydrogen. Crystalline acetyl derivatives of the non-saponifiable vitamin fraction were prepared without destruction of the vitamin.—G. B.

*Orange juice; Commercial drying of — retaining its antiscorbutic properties.* J. F. McClendon and S. M. Dick. *Proc. Amer. Soc. Biol. Chem. J. Biol. Chem.*, 1921, 46, x—xi.

A DEHYDRATING unit consists of an octagonal chamber 20 ft. in diameter and 20 ft. high, with 12 hot-air ports in three tiers. The upper tier admits air at a velocity of 300 ft. per min., the middle at 600 ft., and the lower at 150 ft. per min. A centrifugal spray is in the centre, and just below the level of the upper tier of ports. It consists of a hollow shaft (admitting the juice) and a head made of a series of concave discs serrated at their margins (12 in. in diameter). Between the discs is a series of grooved collars distributing the juice from the hollow shaft to the concave sides of the discs. The shaft rotates at 5000 revolutions per minute and transforms the juice into a fog. The air currents retard the falling of the spray. The exhaust ports are at the bottom of the cell and so large as not to retard the falling of the dried juice in the dead air space. The incoming air is at 55°—70° C., but evaporation prevents the juice reaching this temperature; moreover, a droplet is only exposed for 1 min. to hot air. Orange juice is one-eighth solids; on evaporation to one-fifth of its original weight the pectin forms a clot from which a syrup oozes, but on evaporation to one-sixth no clot separates. The high content of monosaccharides makes the dried juice hygroscopic. The taste is not affected by drying. Milk may be dried in the same apparatus and then mixed with the orange preparation to restore the antiscorbutic substance lost in pasteurisation.—G. B.

*Antiscorbutic property of some desiccated fruit juices.* M. H. Givens and I. G. Macy. *Proc. Amer. Soc. Biol. Chem. J. Biol. Chem.*, 1921, 46, xi—xii.

THE juices of lemons, grapes, tomatoes, raspberries, grape fruit, and orange, dried by the Merrell-Soule Co., Syracuse, N.Y., were examined for antiscorbutic substance in regard to guinea-pigs; all except dried grape and raspberry juices still contained a significant amount; in most cases the products were tested 14—20 months after desiccation. Orange juice was still active when tested two years after being desiccated.—G. B.

*Available carbohydrate in thrice boiled vegetables.* L. O'Reilly and E. H. McCabe. *J. Biol. Chem.*, 1921, 46, 83—89.

BOILING vegetables three times has been advocated in order to remove carbohydrate for diabetics. After such treatment canned string beans, cauliflower, pumpkin, cabbage, and carrots still retained 0.5% of carbohydrate as determined by means of taka-diastase. Boiling with 0.05—0.1% sodium carbonate is much more efficient.—G. B.

*Sea water; Method for utilisation of the nutritive salts of —.* J. F. McClendon. *Proc. Amer. Soc. Biol. Chem. J. Biol. Chem.*, 1921, 46, xxvii.

OCEAN water is treated with phosphoric acid (6g. per l.) and evaporated to complete dryness, giving a non-hygroscopic, non-bitter product containing iodine for the thyroid, fluorine for the teeth, etc. The product may be added to common salt to prevent caking and improve the nutritive qualities of the latter.—G. B.



**Feeds: The salt or sodium chloride content of —.**  
G. S. Fraps and S. Lomanitz. Texas Agr. Exp. Stat., Bull. No. 271, 1920. 14 pages.

To determine the quantity of added salt in fodders, 5.85 g. of the sample is treated with sufficient water to make up 200 c.c. and the mixture shaken for 1 hr.; 80 c.c. of the solution is treated with 1 g. of carbon black or 1 g. of lead acetate, filtered, and 50 c.c. of the filtrate is acidified with nitric acid, an excess of  $N/10$  silver nitrate solution is added, and the excess then titrated with  $N/10$  thiocyanate solution, using ferric alum as indicator. All unmixed fodders contain but small quantities of sodium chloride, with the exception of alfalfa (lucerne) (1.0%), molasses (1.12%), and some meat products (1.3–2.8%).—W. P. S.

See also pages (A) 287, *Evaporation etc.* (Sartory and others). 314, *Purifying proteins* (Field). 316, *Nutritional requirements of yeast* (Nelson and others); *Vitamins and yeast growth* (Williams). 321, *Alkylamines* (Woodward and Alsberg).

#### PATENTS.

*Oxidising nitrogen [e.g., for use in flour bleaching]; Electrodes for —.* F. H. Loring. E.P. 159,709, 31.12.19.

A FLAMING continuous arc is drawn out by means of reciprocating metallic electrodes in a magnetic field, the main axis of which coincides with the axis of the electrodes. For this purpose the lower ferromagnetic electrode or an extension thereof is preferably surrounded by a magnetising coil. The upper electrode may be of copper in tubular form or solid, and in the former case air may be drawn through the tube and passed through the arc. The electrode chamber may be of cast iron to serve as return circuit for the magnetic lines.—J. S. G. T.

*Bread-making.* C. J. Patterson and R. W. Mitchell. U.S.P. 1,370,354, 1.3.21. Appl., 17.11.19.

THE moisture content of dough of a given consistency is increased by the addition of calcium peroxide, not exceeding 0.007% of the weight of flour.—C. A. K.

*Milk food products; Manufacture of —.* C. S. Townsend. E.P. 180,234, 10.12.19.

SKIM milk and vegetable fatty matter each warmed to 75°–100° F. (24°–38° C.) are agitated together, the mixture is emulsified and allowed to stand until lactic acid ether is produced, whereupon the mass is concentrated or dried.—H. H.

*Buttermilk; Condensed —, buttermilk powder, and process for making same.* I. S. Merrell, Assr. to Merrell-Soule Co. U.S.P. 1,370,828, 8.3.21. Appl., 8.4.16.

SEE E.P. 111,340 of 1916; J., 1918, 37 A.

*Alkali phosphates.* G.P. 330,342–3. See VII.

*Caffeine from coffee beans.* E.P. 144,998. See XX.

### XIXb.—WATER PURIFICATION; SANITATION.

*Ice; Water softening for the manufacture of raw water —.* A. S. Behrman. J. Ind. Eng. Chem., 1921, 13, 235–237.

DISSOLVED solids and impurities in natural waters have a detrimental effect on the quality of the ice prepared from them. Calcium and magnesium compounds, present as "temporary hardness," form milky patches and bubbles, and frequently an opaque under crust, which is deposited as a sediment on melting. Permanent hardness is less objectionable, especially if magnesium compounds, which form white ice, are replaced by calcium com-

pounds. Traces of iron give ice a reddish-brown colour; a muddy or bright yellow colour results from the presence of organic matter; and silica and alumina give the ice a muddy appearance and form a slimy sediment on melting. Sodium salts are deposited as white solids and tend to cause brittleness and cracking. During the process of freezing with air agitation most of the above solids collect as a central "core," and may to a certain extent be removed by suction pumping, followed by introduction of fresh water; this, however, involves considerable labour and expense. A satisfactory method of treatment is found in softening the water with lime; calcium chloride and sulphate retained in the water are not appreciably detrimental, and in any case less so than sodium salts; for this latter reason lime is preferable to soda ash. The objections attaching to sodium salts also render the use of zeolites for softening undesirable.

—W. J. W.

*Anthrax spores; Resistance of — to chlorine, pickling liquors, formaldehyde, and mercuric chloride.* A. Müller. Arch. Hyg., 1920, 89, 363–372. Chem. Zentr., 1921, 92, I., 501.

A 1.5% CHLORINE solution killed the spores in 8 hrs., 0.5% solution in 21 hrs. A pickle containing 0.5% of hydrochloric acid was ineffective even after 31 days, 4% hydrochloric acid solution at 37° C. killed the spores after 2 days, 2% hydrochloric acid after 6 days, 1% and 0.5% after 7 days, 0.5% formaldehyde solution killed the spores in 6 days, 5% in 5 days; 0.1% mercuric chloride solution was ineffective in 1 day, and 2% solution in 88 days. At 37° C. 0.1–5% solution of mercuric chloride killed the spores in 20–5 days.—D. W.

*Sulphiformin or methanal-sulphurous acid.* P. Malvezin. Bull. Assoc. Chim. Sucr., 1921, 38, 210–220.

"SULPHIFORMIN" or formaldehyde-sulphurous acid,  $\text{HO.CH}_2\text{SO}_2\text{H}$ , may be prepared by acting on 40% formaldehyde solution with gaseous or dissolved sulphur dioxide, or in a more concentrated form by the interaction of gaseous sulphur dioxide with moist formaldehyde vapour in a suitable apparatus. Various reactions of the product are described, but in a footnote it is stated that the substance first formed on mixing solutions of formaldehyde and sulphurous acid is formaldehyde sulphonylate,  $\text{HO.CH}_2\text{SO}_2\text{H}$ , and this is slowly oxidised to formaldehyde-sulphurous acid. Sulphiformin is readily resolved into its components and accordingly exerts a strongly antiseptic action. A 1% solution has been employed for spraying vines, and experiments described illustrate its toxic action on yeast and bacteria.—J. H. L.

*Nitrous fumes in air.* Moir. See XXIII.

*Adsorption by asbestos.* Koltzoff. See XXIII.

#### PATENTS.

*Water-treating apparatus.* J. Roebe and R. J. Parker, Assrs. to Electric Twin Water Purification Corp. U.S.P. 1,371,814, 15.3.21. Appl. 7.2.19.

AN apparatus for water treatment comprises a container which is divided into compartments by a series of perforated partitions of electrically conducting material. The compartments contain a filtering agent consisting in alternate compartments of a layer of carbon with a superposed layer of quartz and of quartz alone. Means are provided for connecting alternate partitions with the opposite poles of an electric circuit.—W. J. W.

*Distilled water; Process for producing —.* B. Hilliger. G.P. 329,264, 27.5.19.

STEAM is distilled from water at less than atmo-



spheric pressure, and is condensed in a chamber surrounding the distillation vessel, whereby the latent heat evolved on condensation is utilised in heating the water remaining in the distillation vessel. The apparatus is very compact, rendering conduction and radiation losses extremely small.

—J. S. G. T.

Filter-elements. E.P. 134,228. See I.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Yohimbine; Determination of — in yohimbé bark.* A. Schomer. Pharm. Zentralh., 1921, 62, 169—171.

FIFTEEN g. of the powdered bark is shaken for 10 mins. with 150 g. of ether, 10 g. of 15% sodium hydroxide is then added, and, after 1 hr., 100 g. of the ethereal layer is drawn off and extracted with successive quantities (20, 10, and 10 c.c.) of 1% hydrochloric acid; the acid extracts are shaken with chloroform to remove impurities, then rendered alkaline with sodium carbonate solution, and extracted with chloroform. The chloroform extract is evaporated, the residue dissolved in alcohol to which is added a few drops of hydrochloric acid, the alcohol is evaporated, the residue treated with ether and evaporated, and finally warmed with 50 drops of alcohol. When most of the alcohol has evaporated 50 g. of chloroform is added, the mixture cooled at 0° C. for 30 mins., and the crystals of yohimbine hydrochloride then collected, dried at 100° C., and weighed. Three samples of bark examined yielded 0.78—1.26% of yohimbine. The crystals of the hydrochloride obtained had m.pt. 272° C., whilst pure yohimbine hydrochloride has m.pt. 285°—290° C.—W. P. S.

*Humins and humic acids; Synthesis of —.* J. Marcussen. Ber., 1921, 54, 542—545. (Cf. J., 1919, 350 A; 1920, 180 A.)

FURAN when treated with concentrated hydrochloric acid gives a pale brown infusible resin, which is converted by fusion with potassium hydroxide into a humic acid. The course of the reaction appears to be:  $\text{Furan} + \alpha\text{-dihydroxydivinyl, HO.CH:CH.CH:CH.OH} \rightarrow \text{succinaldehyde}$ . The latter yields the aldol and subsequently the  $\alpha$ -peri-difuran, which polymerises with formation of humin. The acidity of the humic acids has been ascribed by Eller and Koch (J., 1920, 717 A) to the presence of phenolic hydroxyl groups; the ready, if incomplete, esterification of these substances when heated with alcoholic hydrogen chloride (3%) and the elimination of carbon dioxide at 250° C. in the presence of superheated steam, however, indicates the presence of carboxyl groups. (Cf. J.C.S., May.)—H. W.

*Yeast-nucleic acid.* S. J. Thannhauser and P. Sachs. Z. physiol. Chem., 1920, 109, 177—182.

No precipitate of the hucine salt of uridinephosphoric acid was obtained by shaking Böhlinger's yeast-nucleic acid with water for 24 hrs., concentrating the filtrate under reduced pressure, boiling with hucine, and treatment with alcohol. It is concluded therefore that the yeast-nucleic acid does not contain free uridinephosphoric acid, and that the barium salt obtained by Steudel and Peiser by fractionating yeast-nucleic acid with barium acetate (J., 1921, 60 A) is not that of uridinephosphoric acid.

*Nucleic acids of the lymphatic ganglions and of the thymus; Physiological properties of the —. Conditions for obtaining a thymonucleic acid very active to blood.* Doyon. Comptes rend., 1921, 172, 820—821.

THE ganglions of the mesentery, carefully freed

from membranes and fat, when treated according to Neuman's method, yield a nucleic acid preparation, which is quite white and permits of the preparation of a very stable, limpid, colourless blood plasma. In preparing nucleic acid from the thymus of a calf, the material should be crushed, mixed with water, and allowed to putrefy for 15—18 hrs. before applying Neuman's method, and only 200—300 g. should be used. This gives a very active preparation.—W. G.

*Arsphenamine [salvarsan]; Arsenical compounds related to —.* G. W. Raiziss and J. L. Gavron. J. Amer. Chem. Soc., 1921, 43, 582—585.

THE undesirable symptoms which sometimes follow intravenous injection of salvarsan are conceivably due to chemical impurities. Some of those possibly present have therefore been examined. 3,5,3',5'-Tetramino-4,4'-dihydroxyarsenobenzene tetrahydrochloride, from the corresponding dinitrohydroxyphenylarsinic acid and sodium hydro-sulphite, exerts a curative influence on trypanosome infections, and exists in two forms, one easily soluble in methyl alcohol, the other sparingly. The preparation of 3,5-diamino-4-hydroxy- and 3-amino-4-hydroxy-phenylarsinic acids (G.P. 224,953; J., 1910, 1178) and the reduction of their acetyl derivatives to arsenobenzene derivatives are described. (Cf. J.C.S., May.)—J. K.

*Neoarsphenamine [neosalvarsan]; Chemistry of — and its relation to toxicity.* G. W. Raiziss and M. Falkov. J. Biol. Chem., 1921, 46, 209—221.

THREE commercial samples were examined, particularly as regards the distribution of the sulphur. A lower arsenic content may be due to the presence of uncombined sodium formaldehyde-sulphoxylate, sulphate, or chloride. The arsenic:nitrogen ratio equals the theoretical in good samples and may be taken as an index of purity. The amount of iodine necessary for complete oxidation is in excess of that required merely by arsenic; this is attributed to uncombined sulphoxylate; sulphoxylate combined with the amino group does not react with iodine. The amount of combined sulphoxylate indicates that the samples were mixtures of mono- and di-substituted products.—G. B.

*Neoarsphenamine [neosalvarsan]; Examination of —.* A. D. Macallum. J. Amer. Chem. Soc., 1921, 43, 643—645.

THE best criterion of the purity of neosalvarsan consists in the determination of the sulphoxylate group,  $\text{CH}_2\text{OSONa}$ , by subtracting from the total reducing power, determined by iodine, that due to free reducing substances and to arsenic, and multiplying the result by 3.9553. Details of the estimations are given, although it is not possible to obtain a product in which the ratio sulphoxylate group:arsenic is exactly 1:2, the best preparations approximate closely to this value.—J. K.

*Adrenaline solutions; Preparation and preservation of —.* F. Richard and M. Malmy. J. Pharm. Chim., 1921, 23, 209—214.

A solution which keeps well is prepared by dissolving 1 g. of adrenaline in 100 c.c. of 0.75% sodium chloride solution containing 1 g. of dissolved sulphur dioxide and then diluting the mixture to 1000 c.c. with 0.75% sodium chloride solution which has been sterilised previously.

—W. P. S.

*Saccharin; Detection of —. A correction.* L. Thevenon. J. Pharm. Chim., 1921, 23, 215.

A REACTION described recently (J., 1921, 60 A) is not characteristic of saccharin, since one of the

reagents used ( $\beta$ -naphthol) itself yields a red coloration with nitrous acid in dilute acid solution.

—W. P. S.

*Aristol.* G. H. Woollett. J. Amer. Chem. Soc., 1921, 43, 553—561.

By the action of one, two, and four equivalents each of iodine and alkali hydroxide on thymol, iodothymol, a gum almost completely soluble in alcohol, and aristol are respectively obtained, and iodothymol can be converted into the other products by treatment with appropriate amounts of iodine and alkali hydroxide. Ordinary aristol is a mixture of the gum (ca. 40%) with a faintly yellow product, insoluble in alcohol, resembling the leuco derivative of Lautemann's Red (Annalen, 1861, 120, 309) in its oxidisability to a red product,  $(C_{10}H_{11}IO)_n$ , sparingly soluble in alcohol. The colour of fresh aristol is due, not to adsorbed iodine, but to this oxidation product, which suffers gradual reduction and liberates iodine from the gummy portion. Lautemann's Red similarly liberates iodine from iodothymol. (Cf. J.C.S., May.)—J. K.

*Dicyanodiamide; Action of sulphuric acid on* —.

T. L. Davis. J. Amer. Chem. Soc., 1921, 43, 669—672.

It is shown that the formation of guanidine by the action of 61% sulphuric acid on dicyanodiamide is slow at 100° C., but an 85% yield is obtained after six hours at 140° C. and the yield is only slightly improved at 200° C. At the higher temperature carbon dioxide is produced in excess of that calculated, probably owing to some further hydrolysis of guanidine. (Cf. J.C.S., May.)—J. K.

*Perchloromethylmercaptan.* O. B. Helfrich and E. E. Reid. J. Amer. Chem. Soc., 1921, 43, 591—594. (Cf. Frankland and others, J., 1920, 256 r).

PERCHLOROMETHYLMERCAPTAN is best obtained by chlorinating carbon bisulphide at 20°–30° C. in diffused light in presence of 0.3–0.4% of iodine until its volume is doubled. Sunlight, a temperature above 30° C., and over-chlorination favour production of carbon tetrachloride, and no other "carrier" than iodine is suitable. By reduction with iron and hydrochloric acid, thiophosgene and carbon tetrachloride are obtained, and the latter is the sole product of boiling the compound with iron alone. Zinc and hydrochloric acid apparently convert it into methane. (Cf. J.C.S., May.)—J. K.

*Nitrotartaric acid.* A. Lachman. J. Amer. Chem. Soc., 1921, 43, 577—581.

NITROTARTARIC acid is the dinitrate of tartaric acid,  $CO_2H.CH(ONO_2).CH(ONO_2).CO_2H$ , since it yields by hydrolysis with fairly concentrated mineral acid nitric and tartaric acids, but with water alone gives rise to nitrous and dihydroxytartaric acids, with some tartronic and oxalic acids, whilst the last two are the only organic acids produced in presence of alkali. In explanation of these reactions, it is suggested that the acid reacts in a pseudo-form,  $CO_2H.C(OH)(ONO_2).C(OH)(ONO_2).CO_2H$ . Although the moist acid decomposes with some violence below 100°, it is stable at the boiling point of xylene when dry. (Cf. J.C.S., May.)—J. K.

[Organic] acids; Identification of —. VI. Separation of acids by means of phenacyl esters. J. B. Rather and E. E. Reid. J. Amer. Chem. Soc., 1921, 43, 629—636.

A NUMBER of illustrations, chosen from naturally occurring organic acids, are given to show that the constituents of a mixture of such acids may frequently be identified by preparation of their

phenacyl esters from the sodium salts (J., 1920, 501 A), followed by fractional crystallisation.—J. K.

*Alkylamines; Detection of volatile* — in the presence of ammonia and volatile tertiary alkylamines in the presence of volatile primary and secondary alkylamines. H. E. Woodward and C. L. Alsberg. J. Biol. Chem., 1921, 46, 1—7.

INCIPIENT decomposition of foodstuffs may be recognised by the presence of traces of amines, which are, however, difficult to distinguish from the ammonia also present. In dilute alkaline solution ammonia reacts with formaldehyde to form hexamethylenetetramine, but methylamines (e.g., 10 c.c. of 0.01–0.02 N amine distilled into 1 c.c. of 40% formaldehyde) furnish methyl alcohol and formic acid. The formic acid is detected by a solution containing 180 g. of mercuric bromide and 120 g. of potassium bromide per litre, which gives on warming a white precipitate of mercurous bromide, when a quantity of formic acid corresponding to 0.5 mg. of amine nitrogen is present. Ammonia, mono- and dimethylamine also give a white precipitate with mercuric bromide, but this is soluble in excess of formaldehyde. Trimethyl and triethylamine form with potassium mercuric iodide (450 g. HgI<sub>2</sub> and 330 g. KI per litre), yellow crystalline compounds melting at 136° and 77° C. respectively, if excess of potassium iodide be avoided. Trimethylamine is still precipitated at 3 mg. in 5 c.c.; dimethylamine requires to be at least six times as concentrated. At suitable concentrations the tertiary amine only is precipitated, and then may be obtained pure by distilling the mercuric iodide compound with sodium hydroxide and sulphide.—G. B.

*Catalytic hydrogenations over copper.* P. Sabatier and B. Kubota. Comptes rend., 1921, 172, 733—736.

THE catalyst is prepared by slowly reducing black tetracupric hydroxide at 200° C. When benzaldehyde and hydrogen are passed over this catalyst at 350° C. the main products are benzene and carbon monoxide, together with some toluene. Under similar conditions acetophenone yields ethylbenzene, and ethyl phenyl ketone yields propylbenzene. Benzoylpropanone is, however, decomposed, giving acetophenone, acetaldehyde, ethyl alcohol, and a little ethylbenzene. Benzoquinone gives almost exclusively quinol. Phthalic anhydride at first yields benzene and carbon monoxide, but soon the action changes and phthalide is formed, together with some toluene and carbon monoxide as products of an accessory change.—W. G.

*Catalysts; Influencing of* — and specifically active catalysts. K. W. Rosenmund and F. Zetzsche. Ber., 1921, 54, 425—437.

THE catalytic reduction of acid chlorides to aldehydes (J., 1918, 412 A) by means of hydrogen in the presence of palladinised barium sulphate is somewhat uncertain, and the effect of a large number of substances on the conversion of benzoyl chloride into benzaldehyde in benzene, toluene, and cumene solution has therefore been investigated. With pure materials, practically no aldehyde is formed; the most effective addition is "sulphured" quinoline (quinoline boiled under a reflux condenser for 5–7 hrs. with one-sixth of its weight of sulphur); specimens of the latter which have been used previously appear to be even better reagents than the freshly-prepared material, the yields reaching 88%. Under similar conditions, o-chlorobenzoyl chloride gives o-chlorobenzaldehyde (more than 70%), p-nitrobenzoyl chloride yields p-nitrobenzaldehyde (91%), and phenylacetyl chloride is converted into phenylacetaldehyde (80%). This appears to be the first recorded instance in which a specific catalyst

has been artificially produced by the addition of a chemically well-defined substance. (*Of. J.C.S., May.*)—H. W.

*Halogen united to nuclear carbon and its replacement by other substituents. III. Preparation of arsinic and sulphonic acids.* K. W. Rosenmund. *Ber.*, 1921, 54, 438–440.

PHENYLARSINIC acid is obtained in small yield when bromobenzene, normal potassium arsenite, water, and copper sulphate are heated at 180°–200° C. Similarly, potassium *o*-bromobenzoate, potassium arsenite, copper powder, and aqueous alcohol give *o*-carboxyphenylarsinic acid at about 90° C. Benzenesulphonic acid is prepared from bromobenzene, sodium sulphite, and copper sulphate in the presence of water at 180° C., and subsequently at 200° C. Similarly, *p*-bromotoluene gives toluene-*p*-sulphonic acid. Sodium *o*-bromobenzoate and sodium sulphite in like manner yield *o*-sulphobenzoic acid at 120°–130° C., or when boiled under a reflux condenser. *m*-Sulphobenzoic acid is prepared from *m*-bromobenzoic acid at 170°–180° C.—H. W.

*Cyanic acid; Synthesis of — by oxidation of organic substances. New methods of detecting this substance.* R. Fosse. *Bull. Soc. Chim.*, 1921, 29, 158–203.

A résumé of work already published: (*cf. J.*, 1919, 655 A; 1920, 747 A, 781 A).—W. G.

**n*-Butyl alcohol and water; Specific gravities of mixtures of —.* Y. D. Wad and A. G. Gokhalo. *J. Ind. Inst. Sci.*, 1921, 4, 17–25.

The specific gravities of mixtures of *n*-butyl alcohol and water covering the entire range of miscibility were determined at temperatures of 20° and 25° C., and from the results curves were made and tables are given showing the specific gravities of aqueous butyl alcohol of all concentrations, from which the following figures are taken:—

% Water.	Sp. gr. at 20° C.	Sp. gr. at 25° C.	% Water.	Sp. gr. at 20° C.	Sp. gr. at 25° C.
0.0 ..	0.8096	0.8066	18.5 ..	0.8448	—
5.0 ..	0.8200	0.8159	94.5 ..	—	0.9886
10.0 ..	0.8292	0.8258	95.0 ..	0.9908	0.9893
15.0 ..	0.8384	0.8349	97.5 ..	0.9942	0.9931
7.5 ..	0.8429	0.8395	100.0 ..	0.9993	—

—G. F. M.

*Aliphatic alcohols; Purification and some physical properties of certain —.* R. F. Brunel, J. L. Crenshaw, and E. Tobin. *J. Amer. Chem. Soc.*, 1921, 43, 561–577.

Owing to their hygroscopic nature it was not found possible to obtain satisfactorily exact boiling points of the alcohols tested, except in presence of drying agents. Of these aluminium amalgam (*J.*, 1895, 898) was the most satisfactory owing to the rapidity of its action; lime and baryta were ultimately equally effective, but metallic calcium, whilst speedy in its action, is not as valuable for this purpose, possibly owing to the presence of an impurity. Boiling points, densities, and refractivities of a number of alcohols are recorded (*cf. J.C.S., May*).—J. K.

*Methyl alcohol; Process for the purification of —.* A. Lanzenberg and J. Duclaux. *Bull. Soc. Chim.*, 1921, 29, 135–136.

COMMERCIAL methyl alcohol usually contains appreciable amounts of acetone in addition to other impurities of higher boiling point. For its purification 1 pt. of alcohol is mixed with 7.5 pts. of chloroform, the mixture distilled, and the fraction boiling at 52.5°–53.5° C. collected. From this fraction the alcohol is extracted with water, the aqueous

alcohol being subsequently distilled and rectified in the usual manner.—W. G.

*Pepsin.* Brewster. *See XIXA.*

#### PATENTS.

*Immunising serum; Process for purifying and enriching —.* Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 104,688, 7.3.17. *Conv.*, 7.3.16.

SERUM which has been freed from euglobulin and electrolytes, preferably by an electro-osmotic process, is placed in the middle chamber of a three-cell electrolytic apparatus; the anodic diaphragm consists of an animal substance, such as hide or bladder, which behaves positively, and the cathodic diaphragm of a vegetable material, such as parchment paper, which behaves negatively to the electric current. The albumins migrate to the anode chamber, whilst the fatty substances separate in the middle chamber. The serum obtained is clear and may be evaporated under reduced pressure to dryness or to any desired concentration. The electric current is applied preferably at high tension and by previous treatment with a relatively low tension it is possible to eliminate the mineral constituents and globulins in the same apparatus.—D. F. T.

*Dialkyl sulphates; Manufacture of —.* L. Lilienfeld. E.P. 143,260, 14.5.20. *Conv.*, 15.12.13.

DIALKYL sulphates are prepared by the direct action of sulphur trioxide on aliphatic alcohols in presence of dehydrating agents, such as phosphorus pentoxide or anhydrous sodium sulphate or copper sulphate, and in presence or absence of inert diluents, such as chloroform, carbon tetrachloride, and the like.—J. H. L.

*Caffeine; Extraction of — from coffee beans.* H. C. E. Lombaers. E.P. 144,998, 19.3.20. *Conv.*, 13.6.19.

In order to avoid condensation of steam and consequent depreciation of the coffee beans during the preliminary "opening up" before the removal of caffeine, the beans are previously raised to the steam temperature. The subsequent steam treatment occupies about 3 hrs., and causes no loss of the valuable extractable and aromatic constituents. After the extraction of the caffeine the beans are again heated before the application of steam for the removal of the extraction agent.—D. F. T.

*Methane; Manufacture of —.* Farhw. vorm. Msister, Lucius, und Brüning. E.P. 146,114, 22.6.20. *Conv.*, 1.7.19. *Addn. to* 146,110 (*J.*, 1921, 26 A).

In the catalytic reduction of carbon monoxide to methane by means of hydrogen, a product of greater purity and free from hydrogen and carbon monoxide is obtained by using carbon dioxide in the later stages of the reaction in the proportion of somewhat more than 1 vol. to every 4 vols. of hydrogen present. Nearly all the hydrogen is thereby converted into methane and water, the latter being periodically separated, whereas with carbon monoxide deposition of carbon commences when the proportion of hydrogen is allowed to become less than 5 vols. for every vol. of monoxide present. The excess of carbon dioxide is finally removed from the product by absorption by lime or other means, and traces of hydrogen by passing over heated copper oxide.—G. F. M.

*Tobacco; Process for the treatment of —.* O. Braemer. E.P. 160,128, 25.10.20.

Tosacco is impregnated with a solution of caffeine.—D. F. T.

**1-Methyl-2-amino-4-isopropyl-5-nitrobenzene.** C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,923, 2.9.19. Appl., 22.9.17.

CYME is treated with a mixture of sulphuric and nitric acids and the nitrocymene reduced to 1-methyl-2-amino-4-isopropylbenzene. This is converted into the 2-acetamino derivative, which on nitration and hydrolysis yields 1-methyl-2-amino-4-isopropyl-5-nitrobenzene, or, if reduced before hydrolysis, the 2,5-diamino compound.

**Dimethyl-di-isopropylbenzidine and process of making same.** C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,924, 2.9.19. Appl., 15.9.17.

CAUDE mononitrocymene is reduced in alkaline solution to hydrazocymene, which when subjected to the benzidine transformation (treatment with hydrochloric acid) is converted into dimethyl-di-isopropylbenzidine, b.p. 250° C.

**Methyl-amino-isopropylbenzenesulphonic acid and process of making same.** C. E. Andrews, Assr. to The Selden Co. U.S.P. 1,314,927, 2.9.19. Appl., 14.9.17.

AMINOCYME is heated with 98% sulphuric acid for 10 hrs. at about 200° C., the product is neutralised with alkali, boiled with charcoal, and precipitated with acid. 1-Methyl-2-amino-4-isopropylbenzene-5-sulphonic acid is obtained.

**Arsenical compound.** W. A. Jacobs, W. H. Brown, M. Heidelberger, and L. Pearce, Assrs. to Rockefeller Institute for Medical Research. U.S.P. 1,315,127, 2.9.19. Appl., 13.6.18.

A SOLUTION OF N-(arsenoaryl)bis- $\alpha$ -aminoacylarylamines (U.S.P. 1,280,123; J., 1919, 268 A) in an alkali hydroxide is evaporated under reduced pressure or precipitated with alcohol to produce alkali salts of, e.g., N-(*p*-arsenophenyl)bis-glycyl-*m*-aminophenol.

$\text{MO.C}_6\text{H}_4.\text{NH.CO.CH}_2.\text{NH.C}_6\text{H}_4.\text{As}$   
 $\text{As.C}_6\text{H}_4.\text{NH.CH}_2.\text{CO.NH.C}_6\text{H}_4.\text{OM}$ ,  
 which are of value in trypanosomal or spirochaetal diseases.

**Organic substances; Process for electrolytic treatment of — without the use of a diaphragm.** Farbenfabr. vorm. F. Bayer und Co. G.P. 310,023, 20.10.16.

THE electrolyte is made up of liquids of different density, one of which contains the organic substance to be treated in solution. Means are provided to prevent the gases liberated at the opposite pole from coming into contact with the liquid containing the substance undergoing treatment. The method may be used for the preparation of piperidine from pyridine and of pinacone from acetone.—A. R. P.

**Organic compounds; Production of — from compounds containing a smaller percentage of hydrogen.** C. Neuberger, A., K., and R. Welde, and E. Heisler. G.P. 330,812, 21.3.14.

REDUCTION of organic compounds, e.g., benzaldehyde to benzyl alcohol, nitrobenzene to aniline, aldol to butyleneglycol, nitromethane or nitroethane to methyl- or ethylamine, or quinone to quinol, is effected by the addition of yeast preparations, or a fermenting mixture of yeast and sugar solution, to the compounds. When reduction is complete, the product is isolated from the mixture.—L. A. C.

**Double salt of glycerophosphoric and lactic acids; Process for the preparation of a —.** E. Merck, Chem. Fabr., and L. Weber. G.P. 331,695, 14.4.20.

CALCIUM or ferric glycerophosphate is allowed to react with the corresponding lactate, and the re-

sulting dark red-brown solution is evaporated at a low temperature to recover the readily soluble double salt. The solutions on boiling do not deposit either calcium or ferric glycerophosphate.—A. R. P.

**Chlorides of aromatic carboxylic acids; Process for the preparation of —.** Badische Anilin- und Soda-Fabr. G.P. 331,696, 17.7.14.

GOOD yields of aromatic acid chlorides are obtained by heating the corresponding trichloride (1 mol.) with water (1 mol.) in the presence of concentrated sulphuric acid and ferric chloride as catalysts. Thus benzotrichloride yields benzoyl chloride, and 2,4-dichlorobenzotrichloride yields 2,4-dichlorobenzoyl chloride as a colourless oil boiling at 152°–155° C. under 30 mm. pressure.—A. R. P.

**Albumins; Combination of metals with —.** K. Kottmann, Assr. to Society of Chemical Industry in Basle. U.S.P. 1,371,380-1, 15.3.21. Appl., 27.9.16 and 26.5.17.

SEE E.P. 110,936 and 115,686; J., 1917, 1289; 1918, 442 A.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Photographic density, light intensity, and exposure time; Relation between —.** F. E. Ross. Communication No. 93 from Research Lab. of Eastman Kodak Co. J. Opt. Soc. Amer., 1920, 4, 255–273.

THE formulae previously suggested by Abney, Hurter and Driffield (J., 1890, 455), Elder, and Channon to express the relation between exposure and image density are examined mathematically and as to their agreement with the experimental values for two emulsions of different type, the one (A) having a high development factor and a short foot, the other (B) having a low development factor and a long foot. Each one is shown to fail on some theoretical consideration and shows also appreciable discrepancy between calculation and experiment, which is more marked with emulsion A than with emulsion B. Taking as the simplest assumptions warranted by available data that the sensitive grains of a plate may be divided into  $n$  groups, each having the same silver content and increasing in sensitiveness by the constant factor,  $r$ , and that each group as a whole obeys the mass action law, the formula deduced is

$$D = d_m \left[ 1 - \frac{1}{n} \sum_{s=0}^{s=n-1} e^{-r^s I t} \right]$$

where  $d_m$  is the maximum density obtainable,  $s$  is the sensitivity factor,  $r$  the common ratio of the sensitivities of the various groups,  $I$  the acting intensity, and  $t$  the exposure time. The method for determining the various constants is developed and calculated compared with experimental values for plates A and B as before; agreement is close for B but not for A. The equations suggested by Schwarzschild [ $I t^n = C$ ] and by Kron

$$(\phi = t \cdot I \cdot 10^{-2} \sqrt{(\log I / I_0)^2 + 1})$$

to express the relation between  $I$  and  $t$  for equal effects on a photographic plate (which does not obey the reciprocity law), are discussed mathematically and some peculiarities deduced which necessitate further experimental work. Assuming Schwarzschild's expression to hold, a method is deduced for determining  $p$  from the values obtained for the development factors for a time scale and an intensity scale exposure.—B. V. S.

*Grain size and sensitometric properties of [photographic] emulsions.* E. P. Wightman and S. E. Sheppard. *Brit. J. Phot.*, 1921, 68, 169—172.

THIS is chiefly a discussion and comparison of papers by Ross (*cf. supra*), Slade and Higson (J., 1921, 27 A), Svedberg (J., 1920, 705 A), and Higson (J., 1921, 98 A). Higson's suggestion that the size of a developed grain is approximately four times that of an undeveloped grain is shown to be inconsistent with data taken from his own and Svedberg's work.—B. V. S.

"*Photographic plate; Action of light on the —, and "The photochemical law of the silver halide grain": a criticism.* L. Silberstein and S. E. Sheppard. *Phot. J.*, 1921, 61, 205—206.

OBJECTION is raised to the conclusion, in the paper by Slade and Higson (J., 1921, 27 A) on which that of Higson (J., 1921, 98 A) is based, that light is not absorbed in quanta by the photographic plate; the statement is based on the observed fact that the rate of conversion of silver bromide grains to the developable condition is not proportional to the light intensity and to the number of grains still unchanged, a fact which has no reference to the quantum theory. It is pointed out that after defining "P," the probability of any grain becoming developable in unit time, from the equation  $dx/dt = P(a-x)$ , it is assumed that P is a function of only I and t and not of x. Further it is stated that the method of arriving at the complete law for the photographic plate by multiplying together the two functions obtained by consideration of the cases when I is variable and t constant and when t is variable and I constant, is mathematically invalid, and the resulting equation is not therefore justified. Other minor criticisms are also put forward.

—B. V. S.

*Colloid chemistry and photography. LII. Acceleration of development by dyes and neutral salts.* Lüppo-Cramer. *Kolloid-Zeits.*, 1921, 28, 174—175.

THE accelerating effect of certain basic dyestuffs on alkaline developers, notably that of Phenosafranine on quinol, has its analogue in a recently observed accelerating effect of certain acid dyes on acid (physical) developers. Thus plates bathed in Erythrosin solution and developed in an acid silver-amidol bath show quicker development; it is preferable, as in all cases of physical development, to treat the plates, before development, in iodide solution which opens up the grain ("lays bare the exposure germ"). Phenosafranine under the same conditions gives a reduction of speed of development. Physical developers are also quickened by addition in considerable concentration of neutral salts such as potassium nitrate and sodium sulphate.

—B. V. S.

*High-speed radiography and radiometallography.* T. Thorne-Baker and L. A. Levy. *Phot. J.*, 1921, 61, 158—166.

By coating a plate first with a special type of light-sensitive emulsion and then with a solution of gelatin containing calcium tungstate in suspension, a composite plate of very high speed to X-rays is obtained. The silver bromide emulsion has as nearly as possible a spectral sensitiveness corresponding with the fluorescence produced by calcium tungstate when excited by X-rays, the spectrograph of which shows maxima at  $\lambda = 24$  to  $25\mu$ ,  $31\mu$ , and  $48\mu$  respectively. The ultra-violet sensitiveness is particularly high and the plate is liable to fog in development when using dark room light filters which are not completely opaque to ultra-violet. The gelatin for the emulsion is specially hardened to stand soaking in water at  $115^{\circ}$ — $120^{\circ}$  F. ( $46^{\circ}$ — $49^{\circ}$  C.) without softening. The calcium tungstate for

the upper film is prepared as an amorphous powder by precipitation, then carefully purified, treated with a very small quantity of impurity, and heated to  $1000^{\circ}$  C. in a carefully controlled electric furnace. The fine crystals which are thus produced are graded, the medium sized ones, having a diameter a little less than that of ordinary silver-bromide plate grains, being used. The calcium tungstate is applied to the light-sensitive film in a solution of a very soft gelatin which easily dissolves in water at  $100^{\circ}$  F. ( $38^{\circ}$  C.). The coating conditions are such that the grains of tungstate settle into close contact with the sensitive film, thus giving maximum efficiency and enabling a small quantity of salt so applied to have as great an intensifying effect as the much larger quantity required for an ordinary intensifying screen. In use the plates after exposure are placed in water at about  $105^{\circ}$  F. ( $40^{\circ}$  C.) which softens and washes away the upper film, the plate being then developed in the usual way, but preferably with an increase of concentration of the developer because of the lower permeability of the film. A hard X-ray tube gives better results and shorter exposures than a soft tube, the full advantage as to speed being obtained with the former only. For this reason the plates are also particularly suitable for radio-metallography.—B. V. S.

*Pinaflavol, a new [photographic] sensitiser for green.* J. M. Eder. *Phot. Kor.*, 1921, 58, 29—31. E. König. *Phot. Rund.* *Brit. J. Phot.*, 1921, 68, Col. Suppl., 16.

PINAFLAVOL (M., L., and B) is one of a new class of basic yellow dyes which are soluble in water and, like Pinacyanol, are stable towards acetic acid but are decolorised by mineral acids. Applied to the photographic plate either by addition to the emulsion or by bathing, Pinaflavol confers a marked sensitiveness in the green and yellow, extending to the D line and practically without the gap in the blue-green shown by plates sensitised by Erythrosin. In three-colour work Pinaflavol plates have the advantage over Pinaverdol plates, in that the relatively sharp ending of the spectral sensitiveness in the yellow enables their use for exposure to green light behind a yellow screen instead of behind a green screen; exposures are considerably reduced because of the greater transparency to green of yellow dyes.—B. V. S.

*Desensitisers.* E. König and Lüppo-Cramer. *Phot. Rund.*, 1921, 37—38.

DYESTUFFS of other classes than the Safranine group (*cf. J.*, 1921, 99 A), containing several amino groups, e.g., Chrysoidine, are somewhat active as desensitisers; those with only one amino group, such as Congo Red, are quite without action. The replacement of the hydrogen of the amino group by methyl has no effect on the desensitising power.

—B. V. S.

*Desensitisers; New application of —. (Handling of X-ray plates in yellow light without use of a preliminary bath and without addition to the developer).* Lüppo-Cramer. *Phot. Kor.*, 1921, 58, 40—43.

X-RAY plates treated with Phenosafranine or similar desensitiser have their sensitiveness to light reduced much more than that to X-rays. This is explained as being due to the fact that light action occurs only on the surface of the silver bromide grains which is protected in the desensitising process by deposition of the dye, while the action of X-rays occurs throughout the whole grain, the interior of which is not protected by dye deposition. After a treatment with Phenosafranine (1/20000), measurements by E. Mauz showed a reduction of X-ray sensitiveness of 25 to 30% in the neighbourhood of the threshold density values, but practically

no reduction in the more exposed parts, while the reduction of light sensitiveness permitted development by a strong light.—B. V. S.

*Desensitising—an oxidation process.* Lüppo-Cramer. *Phot. Ind.*, 1921, 259.

It is suggested that the action of desensitisers is an oxidising action on the latent image, which is bleached if exposed to light in the presence of certain desensitising dyes. Plates which are uniformly exposed, then dyed and then exposed to a subject, give reversed images showing definite sensitiveness. If silver bromide diapositive plates are exposed, converted into iodide, and then stained, the silver iodide complex is strongly colour-sensitive.—B. V. S.

*Colour-toning and negative intensification; The mordant dye process for —.* F. E. Ives. *Brit. J. Phot.*, 1921, 68, 186—187.

The mordant dye process previously described (*cf. J.*, 1921, 99A) for the production of three-colour transparencies is adaptable to the toning of prints on glass or celluloid but not on paper. The bleaching bath recommended is potassium ferricyanide 5 pts., ammonium bichromate 1 pt., glacial acetic acid 110 pts., and water 13,000 pts. The print is placed dry in the bleaching bath for  $\frac{1}{4}$  to 2 mins., no bleaching action being visible, washed for about 5 mins., and transferred to the dye bath. Suitable dyes are Malachite Green, Safranine, Phenosafranine, Rhodamine, and Auramine, made up to a strength of about 1:17000 in water slightly acidified with acetic acid. The process is also adaptable to negative intensification if suitable non-actinic colours are selected, for which purpose a mixture of Victoria Green and Safranine is recommended.

—B. V. S.

*Silver removal process; New photographic —.* A. Steigmann. *Kolloid-Zeits.*, 1921, 28, 175—176.

SILVER is removed, easily and completely, as the metal, in comparatively pure state, from silver solutions such as used "hypo" baths by the use of hydrosulphite. A litre of used "hypo" requires 6–8 g. of hydrosulphite and about the same quantity of caustic soda, about 4 g. of metallic silver being precipitated. It is preferable to work at 50°–60° C. "Hypo" baths so treated may be used again, since the oxidation of hydrosulphite results in the formation of sulphite and thiosulphate.

—B. V. S.

*Photographic registration of chemical reactions.* Jolibois. See XXIII.

#### PATENTS.

*Coloured pictures; Process for making —.* A. Traube. E.P. 147,005, 8.7.20. Conv., 1.2.16.

A picture consisting of a silver image which has been copper-toned (converted to mixed silver-copper ferrocyanide) is further toned by treatment with a solution of a basic dyestuff, about 5 mins. in a dye bath of 1/1000 strength being generally sufficient; the excess of dye is removed from the gelatin by a short washing in water. The transparency of the dyed copper picture is very high and may be still further increased by the removal of the silver ferrocyanide contained in it by treatment with a weak soda fixing solution. A large number of basic dyes are suitable for use in this process, so that almost any desired tone can be obtained.—B. V. S.

*Photographic developer and method of developing latent images.* A. S. McDaniel and A. H. Nietz, Assrs. to Eastman Kodak Co. U.S.P. 1,370,896, 8.3.21. Appl., 10.4.17. Renewed 4.9.20.

DIAMINOPHENOLSULPHONIC acid, in alkaline solution, is used as a photographic developer.

—B. V. S.

*Photographic film.* P. C. Seel, H. Combs, and R. Kemp, Assrs. to Eastman Kodak Co. U.S.P. 1,370,922, 8.3.21. Appl. 10.5.20.

A FILM of nitrocellulose containing aliphatic alcohols with four or five carbon atoms and castor oil to increase the flexibility is provided with a substratum coating before application of the sensitised coating.—B. V. S.

*Negative tracing paper [blue print paper]; Process for improving the keeping quality of —.* E. Merck, Chem. Fabr., and L. Weber. G.P. 331,745, 10.2.20.

AN indifferent salt, such as acid ammonium oxalate or acid ammonium phosphate, which is neither light-sensitive nor produced as a consequence of the reactions following light exposure, is added to the sensitising ferric oxalate preparation.—B. V. S.

*Photographic material; Colour-sensitised —.* F. F. Renwick and O. Bloch, Assrs. to Ilford, Ltd. U.S.P. 1,372,548, 22.3.21. Appl., 21.11.18.

See E.P. 133,770 of 1918; J., 1919, 926A.

## XXII.—EXPLOSIVES; MATCHES.

*Detonation; Transmission of —.* E. Kayser. Z. ges. Schiess- u. Sprengstoffw., 1921, 16, 9–10, 25–27, 33–35.

IN tests for determining the transmission of detonation by air or various solid surfaces, the following results were obtained with a safety explosive consisting of ammonium nitrate, 81%; trinitrotoluene, 17%; and flour, 2%: in air, 6 cm.; on hard ground,  $7\frac{1}{2}$  cm.; on a 10 mm. iron plate, 8 cm.; on loose sand,  $8\frac{1}{2}$  cm.; and in an iron tube of 37 mm. int. diam. and 2.5 mm. thickness, buried in the ground, 60 cm. The influence of the position of the detonator in the charge was illustrated by firing three charges in which the detonator was placed at the end remote from the test charge, in the centre, and at the end adjacent to the test charge, respectively; detonation was communicated at distances of 7, 5, and 3 cm. Although detonators do not transmit the detonation shock beyond  $\frac{1}{4}$  cm., their own sensitiveness to detonation was evidenced by placing them concentrically at different distances from a vertical cartridge with their open ends towards it; positive results were obtained at a distance of 66 cm. The application of the formula  $d = m\sqrt{P}$  ( $d$  is the distance through which detonation is transmitted,  $P$  is the mass of the explosive, and  $m$  is a constant) for measuring the intensity of transmitted shock in the case of cartridges used in practice was investigated in eleven tests each with cartridges of different dimensions and weights. The distance through which transmission is effective, measured from the centre of gravity of the cartridge, is proportional to the length and diameter of the charge. The average value for  $m$  was found to be 1.230. Although reduction of sensitiveness of an explosive both to the initial detonation impulse and to mechanical shock is accompanied by reduced power of transmission, increased sensitiveness does not necessarily imply readier detonation or improved propagation. Thus, addition of charcoal to ammonium nitrate and TNT explosives, or of calcium silicide to ammonium nitrate and dinitronaphthalene explosives, increases the sensitiveness to the falling weight, but causes increased inertness to detonation and reduces the distance through which the detonation shock is transmitted.—W. J. W.

*Potassium chlorate.* Wolcott. See VII.



## PATENTS.

*Explosives.* E. von Herz. E.P. 145,791, 2.7.20. Conv., 25.1.19.

CYCLOTRIMETHYLENETRINITRAMINE prepared by nitration of a cyclotrimethylenetriamine derivative, specially hexamethylenetetramine or cyclotrimethylenetriethyltriamine, is a white, crystalline powder, m.p. 200° C., sp. gr. 1.82, insoluble in water, slightly soluble in alcohol, and more readily soluble in acetone and glacial acetic acid. It is stated to have exceptionally marked explosive properties, whilst being at the same time non-sensitive to shock and friction, and stable even at high temperatures. Its velocity of detonation at maximum density is 8500 m.p.s.; heat of explosion, 1597 cal. per kg.; volume of gas liberated, 905 l. per kg. Either alone, or mixed with other explosives or constituents, it may be used for bursting charges for projectiles, for blasting purposes, or in detonators and fuses.—W. J. W.

*Explosives; Process of producing desensitised — and products thereof.* K. A. Gillespie, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,370,015, 1.3.21. Appl., 3.6.18.

AN explosive contains undried nitrostarch which is desensitised by the presence of water and an oil.—W. J. W.

*Explosive, and process of making explosives.* J. Barab, Assr. to The Commercial Research Co. U.S.P. 1,371,215, 15.3.21. Appl., 14.7.17.

A LIQUID explosive, which is insensitive to friction, consists of an olefine glycol dinitrate.—W. J. W.

*Fertilisers from ammonium nitrate explosives.* G.P. 329,960. See XVI.

## XXIII.—ANALYSIS.

*Platinum resistance thermometers and immersion heating coils of low lag; Construction of —.* T. S. Sligh, jun. J. Amer. Chem. Soc., 1921, 43, 470—475.

CHANCES in the construction of the strain-free type of platinum resistance thermometer from that described in U.S. Bureau of Standards Bull. 6, 150 (J., 1910, 205), consist in the sealing of the thermometer tube to prevent condensation of atmospheric moisture in the thermometer at low temperatures, the use of a simpler thermometer head composed of a brass shell with hakelite base and cap, and the use of thermometer leads consisting of a 4-strand cable insulated with a double silk wrapping and a silk braid, each strand consisting of three No. 28 insulated copper wires twisted together and covered with silk. Calorimetric thermometers of the type described in Bull. 9, p. 483 (1913), have been found to show low values of the  $\delta$  constant in the Callendar difference formula, leading to slight errors in calibration. This is due to a strained winding. The strain may be avoided by thoroughly annealing before winding the coil, careful winding and flash-annealing afterwards. The  $\delta$  value of commercial thermometers can be raised from 1.44 to 1.50 by flash-annealing without opening the thermometer case. An all-metal thermometer case of German silver is described, and also a convenient type of heating coil for laboratory use, made by winding resistance ribbon on a flat strip of mica, insulated by means of thin mica side plates, and enclosed in a flattened copper tube or sheath.—E. H. R.

*Simmanee total-heat recording calorimeter; Report on the —.* T. Gray and A. Blackie. Fuel Research Board, Tech. Paper No. 2, 1921, 23 pages.

FOR a period of five months a Simmanee total-heat recording calorimeter was supplied with town gas, and the readings of the recorder compared with the value of the calorific power of the gas as determined by means of the calorimeter forming part of the instrument. Occasional adjustments of the recorder pen were made, and it was found that the recorded values usually differed from the directly determined values by less than 10 B.Th.U. and seldom by as much as 15 B.Th.U. On two occasions when a greater difference than this occurred, the discrepancy was traced to the accumulation of oxidised metal on the top of the gauze of the burner. The calorimeter was later supplied with various mixtures of coal gas, water-gas, and hydrogen ranging in sp. gr. (air=1) from 0.303 to 0.528, and in calorific value from 326 B.Th.U. to 514 B.Th.U. The greatest difference between the recorded calorific value and that determined by an independent calorimeter was 16 B.Th.U. With the calorimeter run on homogeneous gas, periodic variations occurring at intervals of about an hour were observed, and were due to the accumulation and subsequent sudden removal of air bubbles in the water circuit of the calorimeter. Occasional irregularities of the order of 20 B.Th.U. were attributable to restriction of the water flow by particles of rust etc. Experiments made in order to ascertain the origin of regular differences of the order  $\pm 10$  B.Th.U. showed that the variations of recorded calorific values were roughly parallel with the variations in the gas factor for correcting the gas volume to 60° F. and 30" pressure; that the rate of flow of gas through the meter varied proportionately with the gas factor, and that the claim that the gravity meter automatically corrects the flow of gas for varying pressure and density is substantially true; that variations of the external temperature caused a variation of the recorded calorific value amounting to  $\pm 1.4$  B.Th.U. per  $\pm 1^\circ$  F.; and that owing to the change of viscosity of water with temperature, a change of  $\pm 1^\circ$  F. in the water supply produces a total effect on the recorder reading amounting in some cases to nearly  $\pm 1\%$ .—J. S. G. T.

*Photometric methods and apparatus for the study of colloids.* S. E. Sheppard and F. A. Elliott. J. Amer. Chem. Soc., 1921, 43, 531—539.

TWO types of photometer, distinguished as vertical and horizontal types, for the study of colloids, are described. The vertical type can be modified for use as a nephelometer, colorimeter, microphotometer, dispersimeter, or turbidimeter. The horizontal type is a transmission and scatter photometer, chiefly of use for determining size of particles, comparative turbidities, and coagulation velocities.—E. H. R.

*Photographic registration of chemical reactions accompanied by a variation of pressure; Method of —.* P. Jolibois. Comptes rend., 1921, 172, 809—811.

THE apparatus, which is figured and described, is a simple adaptation of the double galvanometer of Le Chatelier and Saladin for the measurement of pressures in function of temperatures. It permits of the registration in one operation of the vapour pressure of a substance or the dissociation pressure of a compound. Velocities of reaction can be compared and, for a given velocity of heating, the temperature at which a reaction assumes an appreciable velocity can be determined.—W. G.



*Adsorption; Importance of — in analytical chemistry. VIII. Adsorption by asbestos.* I. M. Kolthoff. *Pharm. Weekblad*, 1921, 58, 401–407.

IMPURE asbestos adsorbs positive ions in accordance with the adsorption law. Lead especially is readily adsorbed from dilute solutions, being afterwards easily removed by means of acids; this may be made the basis of a method for the estimation of lead in drinking water. Impure asbestos should be made suitable for analytical work by repeated extraction with acid. (Cf. J.C.S., May.)—S. I. L.

*Potash [; Perchlorate method for the determination of —].* T. D. Jarrell. *J. Assoc. Off. Agric. Chem.*, 1920, 3, 315–321.

THE perchlorate method is accurate and, if a sufficient quantity of perchloric acid is used, the results obtained are not influenced by the presence of phosphates and sodium, magnesium, and calcium salts. Sulphates and ammonia must be removed before the perchloric acid is added. After the potassium salts have been extracted from mixed fertilisers with hot water, the addition of ammonia and calcium oxalate is unnecessary.—W. P. S.

*Calcium group; Simplified methods of analysis in the —.* E. Ludwig and H. Spirescu. *Bul. Soc. Chim. România*, 1920, 2, 35–37.

STRONTIUM is in every case detected by the flame test. After precipitation of the metals of the group with ammonium carbonate in the presence of ammonium chloride the filtrate will contain sufficient calcium and barium, if these elements are present, to permit of their detection with ammonium oxalate and sulphuric acid respectively. Alternatively ammonium oxalate is used as the group precipitant. The filtrate will then contain sufficient barium for its detection with sulphuric acid, and calcium may be detected microchemically in the precipitate by treatment with sulphuric acid. (Cf. Behrens-Kley, *Microchem. Analyse*, 1915.)—W. G.

*Calcium and magnesium; Determination of — in different saline solutions.* E. Canals. *Bull. Soc. Chim.*, 1921, 29, 152–153. (Cf. J., 1920, 283 A, 706 A.)

MAGNESIUM is not precipitated as oxalate along with calcium oxalate if the dilution of the magnesium ion is greater than 1%. Any magnesium which is precipitated, owing to higher concentrations, may be removed by repeated washing with boiling water, it being necessary in some cases to use 500 c.c. of water.—W. G.

*Ferric, aluminium and chromium hydroxides; New method for the separation of —.* M. and M. Lemarchands. *Ann. Chim. Analyt.*, 1921, 3, 86–87.

A MIXTURE of the freshly-precipitated hydroxides is washed until free from ammonia, and then boiled with 10% sodium hydroxide solution to which sodium perborate is added; the chromium and aluminium hydroxides dissolve as sodium aluminate and sodium chromate, respectively, whilst the ferric hydroxide remains insoluble. The latter is separated by filtration; on boiling a portion of the filtrate with the addition of an excess of ammonium chloride, the aluminium hydroxide is re-precipitated. The chromium may be precipitated from another portion of the filtrate as lead chromate.—W. P. S.

*Nickel; Cyanometric assay of —.* G. H. Stanley. *J. S. Afr. Assoc. Anal. Chem.*, 1921, 4, 10–12.

THE nickel solution is rendered slightly ammoniacal, a known amount of silver nitrate (allowance is made for this subsequently) and a small quantity of potassium iodide are added, and the mixture is titrated

with standardised cyanide solution until the precipitate is dissolved completely. If iron is present, the nickel solution is treated with an excess of the cyanide solution, the required amount of ammonia is added rapidly, the mixture filtered, and the excess of cyanide titrated in the filtrate, the nickel being taken by difference. Copper must be removed previously, zinc is eliminated by adding sodium carbonate instead of ammonia to the solution already treated with cyanide, whilst the interference of manganese can be prevented by using ammonium carbonate in place of ammonia. Stannic compounds do not interfere.—W. P. S.

*Cobalt; New method for the detection and determination of —.* S. A. Braley and F. B. Hobart. *J. Amer. Chem. Soc.*, 1921, 43, 482–484.

SOLUTIONS containing cobalt give, with a solution of dimethylglyoxime containing ammonia or sodium acetate, a brown coloration which is characteristic for cobalt inasmuch as, unlike the coloration given by other metals, it is not discharged by mineral acids. In absence of copper and iron it provides a sensitive qualitative test for cobalt. The colour can also be made to serve for a colorimetric estimation of cobalt in presence of nickel. The solution, prepared as for the determination of nickel, is slightly acidified, treated with sodium acetate, heated to boiling, and then the dimethylglyoxime added in slight excess. The nickel precipitate is filtered off, the solution cooled slowly, and when cold compared colorimetrically with standards.—E. H. R.

*Mercury and copper; Electrolytic separation of —.* W. Böttger. *Z. angew. Chem.*, 1921, 34, 120–122.

IN PRESENCE of chlorine ions the separation of mercury from copper is most effectively achieved by first removing both metals from the solution at a sufficiently high voltage, e.g., 2.2–2.4 volts. They are then dissolved in dilute nitric acid, and the solution, after neutralisation and addition of acid and alcohol, is electrolysed at 1.4 volts. When potassium cyanide is added to a mercury-copper solution containing chlorine ions, the addition of a few c.c. of concentrated ammonia is advantageous in preventing decomposition of the cyanide. Separation of copper from such a solution takes place at normal temperature with a voltage of 3.2 volts; without ammonia the necessary voltage is 3.8–4.5 volts. At 65° C., an appreciably lower voltage, e.g., 2 volts, may be used. (Cf. J.C.S., May.)—W. J. W.

*Uranium, vanadium, and chromium; Qualitative separation and detection of — when present together.* P. E. Browning. *J. Amer. Chem. Soc.*, 1921, 43, 114–115.

A SOLUTION containing chromic and vanadic acids and a uranium salt is made faintly alkaline with ammonia and then faintly acid with acetic acid and treated with ammonium phosphate. Uranium phosphate is precipitated, filtered off, and washed. The identity of the uranium phosphate is confirmed by the addition to the precipitate of a drop of potassium ferrocyanide, when a red-brown stain is produced. The filtrate is treated with excess of sulphur dioxide to reduce the chromic and vanadic acids; the excess of sulphur dioxide is boiled off, an excess of bromine added, and the excess removed by boiling; this oxidises the vanadium to vanadic acid, but does not affect the chromium. Ammonia is added whereby the chromium separates as a green precipitate of phosphate and hydroxide. The filtrate from the chromium is made alkaline and saturated with hydrogen sulphide, which gives the pink or violet oxysulphide of vanadium.—J. F. S.

*Gallium ferrocyanide; Use of — in analysis.* L. E. Porter and P. E. Browning. J. Amer. Chem. Soc., 1921, 43, 111—114.

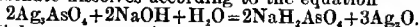
GALLIUM ferrocyanide passes through asbestos or filter paper in a Gooch crucible or filter paper in a funnel when suction is applied, but it can be filtered satisfactorily by using a pad made of shredded filter paper placed on a double filter paper and applying gentle suction. The precipitate after washing is ignited and weighed as the mixed oxides of gallium and iron. Gallium may be recovered from the ferrocyanide by solution in sodium hydroxide and precipitation of gallium hydroxide with carbon dioxide. An alternative method consists in treating the alkaline solution of gallium ferrocyanide with hydrogen peroxide to oxidise the sodium ferrocyanide to ferricyanide and then boiling with ammonium chloride, when pure gallium hydroxide is precipitated. When zinc is present neither method is available. In this case the metals are precipitated together as ferrocyanides which are dissolved in sodium hydroxide and the hydroxides precipitated by carbon dioxide. These are dissolved in hydrochloric acid and the gallium precipitated by boiling with ammonium bisulphite. Alternatively the alkaline solution of ferrocyanides is treated with hydrogen sulphide and the zinc precipitated, the gallium then being obtained by either of the methods described above.—J. F. S.

*Nitric nitrogen; Comparative results with Scales' method and Devarda's alloy for reducing —.* A. P. Harrison. J. Biol. Chem., 1921, 46, 53—56.

SCALES' zinc-copper couple gives as accurate results as Devarda's alloy, and has the advantage of not requiring to be weighed. 200 c.c. of solution with 0.1 mg. N per c.c. is poured into a flask containing about 80 g. of freshly prepared zinc-copper couple; about 5 g. of sodium chloride and 1 g. of magnesium oxide are added, and 150 c.c. is distilled into a flask containing 50 c.c. of 4% boric acid. Bromophenol blue is used as indicator in the titration.—G. B.

*Arsenate and arsenite; Separation and detection of —.* G. W. Sears. J. Amer. Chem. Soc., 1921, 43, 466—470.

DETERMINATIONS of the solubility of silver arsenate and silver arsenite respectively in sodium hydroxide solution show that, whereas the former is readily dissolved in the cold by 0.5 to 1.5N sodium hydroxide, the latter is practically insoluble. The arsenite dissolves according to the equation



Higher concentrations of sodium hydroxide react slowly with silver arsenite with formation of arsenate and metallic silver. To separate arsenate and arsenite, their nitric acid solution containing excess of silver nitrate is neutralised with sodium hydroxide until the silver oxide precipitate just fails to re-dissolve. The washed precipitate is then shaken with about 25 c.c. of a solution containing about 3 c.c. of 6N sodium hydroxide solution. This is sufficient to dissolve at least 0.45 g. of silver arsenate. The arsenate and arsenite can be detected in the filtrate and residue respectively by usual methods.—E. H. R.

*Air; Determination of nitrous fumes in — with special reference to fuse-igniters.* J. Moir. J. S. Afr. Assoc. Anal. Chem., 1921, 4, 3—7.

A METRON, similar to that of Hoesvay, but applicable to both small and large amounts of nitrite or nitrous acid, is described. The liquid under examination is treated with 1—10 c.c. of *p*-nitraniline solution (*p*-nitraniline, 1.5 g., concentrated hydrochloric acid, 10, water to 500 c.c.) and heated at 50° C.; 1—10 c.c. of  $\alpha$ -naphthol solution ( $\alpha$ -naphthol, 2, sodium hydroxide, 1.1, sodium acetate, 10 g.,

water to 500 c.c.) is then added and, after 1 hr., the orange-red precipitate is collected and weighed; 92 g. of  $\text{NO}_2$ , or 69 g. of  $\text{NaNO}_2$ , yields 293 g. of precipitate. The latter dissolves in sodium hydroxide solution with an intense blue-violet colour, and very small quantities may be determined colorimetrically. To determine  $\text{NO}_2$  apart from nitrite in the fumes from fuse igniters, a definite volume of the gases may be left in contact with neutral hydrogen peroxide solution for 24 hrs., and the solution then titrated, using methyl orange as indicator; 1 c.c. of *N*/10 alkali is equivalent to 0.0060 g.  $\text{NO}_2$ . Alternatively, neutral potassium iodide solution may be used in place of the hydrogen peroxide and the liberated iodine titrated with thio-sulphate solution; each c.c. of *N*/10 thio-sulphate solution = 0.0051 g.  $\text{NO}_2$ .—W. P. S.

*Iodoform reaction; Sensitive modification of the —.* N. Schoorl. Pharm. Weekblad, 1921, 58, 209—210.

AMMONIUM persulphate is used with potassium iodide in alkaline solution. The reaction will detect 1 pt. of acetone in 100,000. (Cf. J.C.S., April.)—S. I. L.

*Miscible liquids; Separation of — by distillation.* S. F. Dufton. Phil. Mag., 1921, 41, 633—646.

THE still-head described previously by the author (J., 1919, 457) gives a complete separation of simple binary mixtures and renders possible the examination of the rationale of separation by distillation. The physical processes occurring in the still-head are discussed mathematically and amplified by experimental data. Comparison of continuous and discontinuous distillation shows that the former is much more economical; further, a column can be employed at its maximum efficiency throughout, whilst the length must be variable if the highest efficiency in discontinuous distillation is to be obtained.—W. P. S.

See also pages (A) 289, *Heat of carbonisation of coal* (Strache and Grau); *Benzene hydrocarbons in gas* (Berl and others). 295, *Oxy- and hydro-celluloses* (Schwalbe and Becker). 297, *Burner gases* (Dieckmann). 298, *Arsenious compounds* (Meurice). 312, *Tannin analysis* (Kubelka and Köhler). 315, *Invert sugar in presence of sucrose* (Kraiszy). 316, *Iron in organic liquids* (Malvezin and Rivalland). 317, *Hydrogen-ion concentration of milk* (Kramer and Greene); *Organic acids in milk* (Kramer and Greene); *Milk* (Reiss); *Moisture in milk powder* (Schoorl and Gerritzen); *Proteins* (Cohn). 318, *Pepsin* (Brewster); *Alkalinity and phosphates in ashes of foods* (Tillmans and Bohrmann). 319, *Salt content of feeds* (Fraps and Lomanitz). 320, *Yohimbine* (Schomer); *Neosalvarsan* (Macallum); *Saccharin* (Thevenon). 321, *Organic acids* (Rafter and Reid); *Alkylamines* (Woodward and Alsberg).

#### PATENTS.

*Gas analysing apparatus.* Aktiebolaget Ingeniörs-firma F. Egnell. E.P. 106,265, 29.9.16. Conv., 8.5.16.

IN gas analysing apparatus provided with an oxidation chamber and a vessel for absorption of carbon dioxide, a cock is arranged between the gas generator and the analysing apparatus and is of such construction that the gas to be analysed can be passed from the generator through the cock to the absorption vessel, thence, through the cock to the oxidation chamber and finally through the cock to the analysing apparatus, or directly through the cock to the latter. If desired, the gas may be led directly from the absorption vessel to the analysing apparatus, and the gas generator may be shut off from the rest of the apparatus. The cock is provided with a device for closing and breaking the electric circuit for heating the oxidation chamber.—J. S. G. T.

*Gases and vapours capable of being absorbed; Continuous determination of —.* Badische Anilin- und Soda-Fabrik. G.P. 329,386, 27.4.18.

THE gaseous mixture is simultaneously delivered through two parallel circuits, each provided with a pair of throttle valves. In the one circuit, absorption of the gas or vapour concerned is effected, and the difference of pressure in the two circuits arising therefrom at constant temperature enables the amount of the absorbed constituent to be ascertained.—J. S. G. T.

*Thermo-element for the measurement of high temperatures.* F. Hesse. G.P. 330,726, 5.3.18.

THE thermo-junction is constituted of tantalum and molybdenum and is fused into a sheath filled with basic refractory material to prevent oxidation.—J. S. G. T.

*Heating-value indicator.* U.S.P. 1,370,945. See II A.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

- Acheson. Deflocculating solid substances. 11,199. Apr. 18. (U.S., 1.5.20.)  
 Akt.-Ges. f. Stickstoffdünger. Treatment of feed-water for steam generators etc. 10,608. Apr. 11. (Ger., 12.4.20.)  
 Beldimano, Robertson, and Stanley. Evaporating or heating liquids. 10,953. Apr. 14.  
 Brown. Carbon bleaching or filtering material, and method of making same. 11,580. Apr. 21. (U.S., 12.8.20.)  
 Chaudière. Furnaces, gas-producers, etc. 11,321. Apr. 19. (Belg., 20.4.20.)  
 Johnston. Machines for centrifugally separating solids from liquids. 10,901. Apr. 14.  
 Leubli. Grinding-mills. 10,963. Apr. 14.  
 Marks (Shell Co.). Re-run still. 11,438. Apr. 20.  
 Mercer. Pulverising or disintegrating machines. 10,634. Apr. 12.  
 Powdered Fuel Plant Co., Ltd. Means for separating suspended solid from a gaseous medium. 11,014. Apr. 15. (Fr., 12.8.20.)  
 Reid. Furnaces. 10,704. Apr. 12.  
 Rigby. Drying-cylinders. 11,278-9. Apr. 19.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 15,265 (1918). Gal. Furnaces. (122,395.) Apr. 20.  
 19,287 (1919). Harris. Drying process and apparatus. (161,598.) Apr. 27.  
 19,639 (1919). Junquera. Manufacture of concentrated solutions. (144,240.) Apr. 27.  
 29,288 (1919). Coleman. Mixing and agitating machines. (161,617.) Apr. 27.  
 169 (1920). Paget. Grinding, refining, or reducing apparatus. (161,257.) Apr. 20.  
 325 (1920). Still. Filters. (161,639.) Apr. 27.  
 1176 (1920). Hirsch. Apparatus for generating gas by the action of liquids on solid reagents. (161,681.) Apr. 27.  
 3707 (1920). Barbet et Fils et Cie. Condensing arrangements of rectifying apparatus. (138,869.) Apr. 20.

- . 5224 (1920). Kennedy. Gyratory crushing apparatus. (139,216.) Apr. 27.  
 5714 (1920). Dorr Co. Rotating screen filtering apparatus. (139,493.) Apr. 20.  
 8943 (1920). Johnson (Fischlein). Centrifugal apparatus for straining liquids. (161,822.) Apr. 27.  
 20,141 (1920). Danhardt. Asbestos cloth, especially for dry separation of solid matter from blast-furnace gases etc. (153,558.) Apr. 20.  
 20,289 (1920). Rudisill. Pulveriser. (148,369.) Apr. 20.  
 24,280 (1920). Hardinge. Grinding and disintegrating mills. (150,997.) Apr. 27.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

- Accioly. Manufacture of an alcoholic liquid (synthetic) gas for use as combustible. 11,419. Apr. 20.  
 Bowler. Distilling crude petroleum. 11,623. Apr. 22.  
 Burnet. Vertical retorts for destructive distillation. 10,834. Apr. 13.  
 Burton. Gas-retort mouthpieces. 11,134. Apr. 16.  
 Carratt. Gas-producers. 11,393. Apr. 20.  
 Chaudière. 11,321. See I.  
 Clapp, and Ferolite, Ltd. Gas-producers, electric furnaces, crucible furnaces, etc. 11,065. Apr. 15.  
 Davidson, and Low Temperature Carbonisation, Ltd. Retorts. 11,222. Apr. 18.  
 Dehn (McComb). Converting heavy liquid hydrocarbons or liquid hydrocarbons of higher boiling point into lighter hydrocarbons or hydrocarbons of lower boiling point. 11,116. Apr. 16.  
 Frentrop, Kiederich, and Meyer zu Eissen. Increasing the consistency of hydrocarbon oils etc. 10,692. Apr. 12.  
 Glawe. Mixing briquette-forming materials with fluid binding agent. 10,919. Apr. 14.  
 Keith, Whatmough, and Keith and Blackman Co., Ltd. Liquid fuel, and process for production thereof. 11,444. Apr. 20.  
 Marks (Shell Co.). 11,438. See I.  
 Powdered Fuel Plant Co., Ltd. Apparatus for pulverising coal etc. 11,012-3. Apr. 15. (Fr., 17 and 31.8.20.)  
 Trent Process Corp. Process of separating oils. 10,828. Apr. 13. (U.S., 11.8.20.)  
 Trent Process Corp. Manufacture of combustible gas. 10,829. Apr. 13. (U.S., 31.8.20.)  
 Trent Process Corp. Distillation of oils. 10,831. Apr. 13. (U.S., 4.11.20.)  
 Wellford. Treatment of hydrocarbons. 11,074. Apr. 16.  
 Wood. Gas-retorts. 11,041. Apr. 15. (Australia, 15.4.20.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 24,047 (1919). Rütgerswerke A.-G. Process of making a substitute for petroleum jelly. (134,528.) Apr. 20.  
 24,852 (1919). U.S. Industrial Alcohol Co. Liquid fuel. (133,709.) Apr. 27.  
 25,104 (1919). Low Temperature Carbonisation, Ltd., Davidson, and Armstrong. Retorts for distilling carbonaceous substances. (161,608.) Apr. 27.  
 32,280 (1919). Rütgerswerke A.-G. Production of soot carbon, retort graphite, and other carbon products from natural gas. (137,065.) Apr. 20.  
 70 (1920). Paris. Cleaning and refining petroleum and analogous distillates. (161,253.) Apr. 20.  
 8797 (1920). U.S. Industrial Alcohol Co. Liquid fuels. (140,797.) Apr. 20.  
 17,612 (1920). Nasspress Ges. Process of drying raw peat. (146,214.) Apr. 20.

18,266 (1920). Bergius and Kalnin. Separate recovery of constituents of the distillation of gas from fuel. (146,332.) Apr. 27.  
 19,518 (1920). Still. Purifying coal gas by means of ammonia. (147,583.) Apr. 27.  
 20,141 (1920). Danhardt. *See I.*

### III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Commin and Hughes. Treatment of pitch. 11,060. Apr. 15.  
 Imray (Soc. Chem. Ind. in Basle). Manufacture of  $\alpha$ -aminoanthraquinone. 11,459. Apr. 20.  
 Koppers Co. Purification of phenol-contaminated liquors. 10,603. Apr. 11. (U.S., 30.4.20.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

4595 (1920). Koppers Co. Purification of liquors containing phenoloid bodies. (139,168.) Apr. 20.  
 14,276 (1920). South Metropolitan Gas Co., and Stanier. Manufacture of certain naphthylamine-sulphonic acids. (161,859.) Apr. 27.  
 19,123 (1920). Meister, Lucius, u. Brünig. Manufacture of pyridine bases. (147,101.) Apr. 20.

### IV.—COLOURING MATTERS AND DYES.

#### COMPLETE SPECIFICATION ACCEPTED.

20,256 (1920). Cassella und Co. Manufacture of a new colour of the anthraquinone series. (148,339.) Apr. 20.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

Bloxam (Technochemia A.-G.). Manufacture of textile products from animal fibres. 11,461. Apr. 20.  
 Courtaulds, Ltd., Callimachi, and Hegan. Manufacture of threads, filaments, etc. of cellulose. 11,766, 11,768-9.  
 Courtaulds, Ltd., and Wilson. Production of coloured threads, filaments, etc. 11,770. Apr. 23.  
 Dreyfus. Manufacture of cellulose derivatives. 11,293. Apr. 19.  
 Krantz. Method of drying textile materials. 11,169. Apr. 18.  
 Plauson's (Parent Co.), Ltd. (Plauson). Manufacture and treatment of viscose. 10,615. Apr. 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

22,680 (1919). Pickup and Wilson. Treating fibres. (161,600.) Apr. 27.  
 27,596 (1919). Pritchard. Degumming and washing fibres. (161,219.) Apr. 20.  
 30,954 (1919). Maupai. Process of treating silks. (161,625.) Apr. 27.  
 694 (1920). Power Gas Corp., and others. *See XVIII.*  
 6520 and 6643 (1920). B. G. Textilwerke Ges. Balloon envelope materials. (139,795 and 139,807.) Apr. 27.  
 17,721 (1920). Soc. Suisse de Ferments. Degumming textile materials. (145,583.) Apr. 27.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### APPLICATIONS.

Courtaulds, Ltd., and Wilson. 11,770. *See V.*  
 Dicker (Bever). Impregnating and dyeing porous materials. 10,613. Apr. 11.  
 Farrell. Dye-jigs or becks. 10,627. Apr. 12.

Henshilwood and Thornber. Machines for bleaching, dyeing, etc. 11,272. Apr. 19.

#### COMPLETE SPECIFICATIONS ACCEPTED.

13,161 (1920). Bayer u. Co. Process for dyeing with acid sulphonamino dyestuffs, and the manufacture of new lakes. (143,242.) Apr. 20.

### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

#### APPLICATIONS.

Allen and Hanburys, Ltd., and Lizius. 11,659. *See XXIII.*  
 Chem. Fabr. Griesheim-Elektron, Pistor, Reitz, and Schultze. Manufacture of stable compounds containing active oxygen and calcium. 10,771. Apr. 13.  
 Constant and Raisin. Production of boron. 11,645. Apr. 22. (Fr., 22.4.20.)  
 Deuts. Gold- u. Silber-Scheideanstalt. Production of sodium cyanide. 11,717. Apr. 22. (Ger., 10.6.20.)  
 Feldenheimer and Plowman. Process for grading alkaline-earth carbonates. 11,358. Apr. 19.  
 Mirat and Pipereant. Manufacture of sulphuric acid. 10,792. Apr. 13. (Belg., 6.5.20.)  
 Pease. Manufacture of compounds of ammonia etc. 11,039. Apr. 15.  
 Riedel A.-G. 11,068. *See XX.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

10,401 (1918). Deuts. Gold- u. Silber-Scheideanstalt. Manufacture of alkali percarbonates. (117,085.) Apr. 27.  
 24,875 (1919). Spence and Sons, Spence, and Llewellyn. Manufacture of aluminous compounds. (161,606.) Apr. 27.  
 30,611 (1919). Patrick. Silica gels and process of making same. (136,543.) Apr. 20.  
 32,756 (1919). Douglas. Manufacture of ammonia. (161,244.) Apr. 20.  
 353 (1920). Kelly. Manufacture of acid sodium pyrophosphate. (161,273.) Apr. 20.  
 822 (1920). Mackay. Obtaining strontium and barium sulphates from their native sulphates. (161,655.) Apr. 27.  
 823 (1920). Mackay. Obtaining copper sulphate direct from metallic copper. (161,656.) Apr. 27.  
 1231 (1920). Wildman. Extraction of alumina and its salts from clay. (161,310.) Apr. 20.  
 10,004 (1920). Clayton. Apparatus for manufacture of oxygen compounds of sulphur. (161,439.) Apr. 20.  
 17,437 (1920). Röhm. Production of a solid non-hygroscopic iron salt. (146,214.) Apr. 20.

### VIII.—GLASS; CERAMICS.

#### APPLICATIONS.

Clark, Taylor, and Universal Transfer Co. Process for pottery printing. 10,741. Apr. 13.  
 Rigby. Treatment of china clay etc. 11,093. Apr. 16.  
 Wallis. Composition for manufacture of crockery-ware. 11,139. Apr. 18.

### IX.—BUILDING MATERIALS.

#### APPLICATIONS.

Brant. Building etc. materials. 10,781. Apr. 13.  
 Leno. Cement brick. 11,574. Apr. 21. (Denmark, 27.4.20.)  
 Miller, Miller, and Williams. Damp-insulating material for buildings etc. 11,249. Apr. 19.

## COMPLETE SPECIFICATIONS ACCEPTED.

13,164 (1920). Wennerstrom. Manufacture of cement. (143,243.) Apr. 27.  
 16,297 (1920). Schiff. Manufacture of dental cements. (161,868.) Apr. 27.  
 17,312 (1920). Larsen. Manufacture of porous cement or mortar products. (146,172.) Apr. 27.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

## APPLICATIONS.

Beilstein, and Chem. Fahr. Griesheim-Elektron. Recovery of light metals from scrap. 11,066. Apr. 15.  
 British Thomson-Houston Co. (General Electric Co.). Electro-metallurgy. 11,049. Apr. 15.  
 Chandler, and South Metropolitan Gas Co. Gas-fired metallurgical furnaces. 11,699. Apr. 22.  
 Clapp and others. 11,065. See II.  
 Coles. Manufacture of bronze powders. 11,520. Apr. 21.  
 Continuous Reaction Co., Merson, and Skelley. Manufacture of ferrotungsten and ferromolybdenum. 11,786. Apr. 23.  
 Continuous Reaction Co., Skelley, and Smith. Manufacture of ferrous alloys. 11,787. Apr. 23.  
 Danforth. Open-hearth furnaces. 11,437. Apr. 20.  
 Hervey and Taylor. Treatment of iron. 10,707. Apr. 12.  
 Rogers (Calorizing Corp. of America). Calorizing furnace and process. 11,310. Apr. 19.  
 Rogers (Calorizing Corp. of America). Metallic alloys. 11,311. Apr. 19.  
 Sweeney. Manufacture of composite metallic sheets. 10,846. Apr. 13.  
 Trent Process Corp. Process of reducing ores. 10,830. Apr. 13. (U.S. 4,10,20.)

## COMPLETE SPECIFICATIONS ACCEPTED.

24,480 (1919). Morgan Crucible Co., and Speirs. Electrically heated melting and other furnaces. (161,603.) Apr. 27.  
 821 (1920). Mackay. Obtaining iron and tin from tin scrap. (161,654.) Apr. 27.  
 3692 (1920). Vigeeon and McConway. Pre-leaching apparatus for treating calcined ore and the like. (161,375.) Apr. 27.  
 4146 (1920). Crosthwaite Eng. and Furnace Co., and Crosthwaite. Furnaces for heating crucibles employed for melting or heating metals etc. (161,386.) Apr. 20.  
 20,141 (1920). Danhardt. See I.  
 23,238 (1920). Tugwood (Dwight and Lloyd Metallurgical Co.). Ore-sintering mechanisms. (161,491.) Apr. 20.  
 658 (1921). Agostini. Solder for aluminium and other metals. (156,665.) Apr. 20.

## XI.—ELECTRO-CHEMISTRY.

## APPLICATION.

Clapp and others. 11,065. See II.

## COMPLETE SPECIFICATIONS ACCEPTED.

24,480 (1919). Morgan Crucible Co., and Speirs. See X.  
 25,052 (1919). Trembour. Electric furnaces. (133,954.) Apr. 27.  
 27,150 (1919). Boorne and Darge. Covering metal cores in the manufacture of electrodes. (161,212.) Apr. 20.  
 66 (1920). Bultemann. Manufacture of an electric insulating material. (137,326.) Apr. 20.  
 29,780 (1920). Smalley. Electric furnaces. (152,686.) Apr. 27.

## XII.—FATS; OILS; WAXES.

## APPLICATIONS.

Eppenberger. Rendering fat-containing granular products impalpable. 10,940. Apr. 14. (Switz., 1.3.21.)  
 Fenton. Treating oil-bearing solids. 10,710. Apr. 12.  
 Kohayashi. Manufacture of hydrocarbons from fatty oils. 11,712. Apr. 22. (Japan, 13.10.20.)  
 Plauson's (Parent Co.), Ltd. (Plauson). Compositions of oils etc. 10,616. Apr. 12.  
 Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of viscous oily compositions. 10,852. Apr. 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

30,543 (1919). Heyerdahl. Extraction and refining of oil. (137,514.) Apr. 20.  
 8354 (1920). Quick. Refining and decolorising animal and vegetable oils. (161,813.) Apr. 27.  
 18,752 (1920). Elektro-Osmose A.-G. Purification of glycerin. (146,865.) Apr. 27.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

## APPLICATION.

Dargan. Luminous ink. 11,163. Apr. 18.

## COMPLETE SPECIFICATIONS ACCEPTED.

17,490 (1919). Ivinson and Roberts. Anti-corrosive paint or composition. (161,201.) Apr. 20.  
 24,916-7 (1919). Melamd. Production of resin-like bodies. (133,712-3.) Apr. 27.  
 469 (1920). Baker. Process of making pigments. (161,280.) Apr. 20.  
 13,161 (1920). Bayer u. Co. See VI.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

## APPLICATIONS.

Catalpo, Ltd., and Schidrowitz. Manufacture of caoutchouc and caoutchouc-like products. 11,055. Apr. 15.  
 Moler. Manufacture of regenerated rubber. 11,053. Apr. 15.

## COMPLETE SPECIFICATIONS ACCEPTED.

748 (1920). Gare. Reforming or vulcanising rubber, ebonite, vulcanite, etc. (161,648.) Apr. 27.  
 799 (1920). Farrel Foundry and Machine Co. Mixing or masticating rubber etc. (137,820.) Apr. 20.  
 4264 (1920). Helhronner. Manufacture of india-rubber products. (142,063.) Apr. 27.  
 6670 (1920). Bruni. Vulcanisation of rubber. (140,387.) Apr. 27.  
 18,916 and 18,920 (1920). Wade (Goodyear Tire and Rubber Co.). Manufacture of rubber products. (161,482-3.) Apr. 20.

## XV.—LEATHER; BONE; HORN; GLUE.

## APPLICATIONS.

Richter. Removal of hairs from hides etc. 10,775. Apr. 13.  
 Soc. Pichard Frères. Process for unhairing hides. 11,638. Apr. 22. (Fr., 12.5.20.)

## COMPLETE SPECIFICATIONS ACCEPTED.

27,772 (1919). Malcolm and Townsend. Manufacture of vegetable gelatin. (161,612.) Apr. 27.  
 2652 (1920). Boys. Apparatus for mixing or agitating lime in tannery pits. (161,362.) Apr. 20.

8681 (1920). Bray (Indian Wood Products Co.). Manufacture of catechin and catechu-tannic acid. (161,431.) Apr. 20.

#### XVI.—SOILS; FERTILISERS.

##### COMPLETE SPECIFICATION ACCEPTED.

4303 (1920). Sams. Fertiliser and process of making same. (161,777.) Apr. 27.

#### XVII.—SUGARS; STARCHES; GUMS.

##### APPLICATIONS.

Brown. Washing starch. 11,581. Apr. 21. (U.S., 12.8.20.)

Courtaulds, Ltd., Lloyd, and Stokes. Production of compounds or mixtures of starch and sulphuric acid. 11,767, 11,782. Apr. 23.

Tiemann. Purifying sugar juices by filtration and decantation. 11,561. Apr. 21. (Ger., 21.4.20.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

1972 (1920). Barbet et Fils et Cie. *See* XIX.

11,691 (1920). Classen. Manufacture of sugar and furfural from wood and other cellulosic substances. (142,430.) Apr. 27.

12,609 (1920). Terrisse and Levy. Obtaining glucose from wood. (143,212.) Apr. 20.

18,744 (1920). Wohl. Saccharification of cellulose-containing materials. (146,860.) Apr. 27.

22,019 (1920). Tavorog, Roche, and Martin. Manufacture of lactose or milk sugar. (161,887.) Apr. 27.

#### XVIII.—FERMENTATION INDUSTRIES.

##### COMPLETE SPECIFICATIONS ACCEPTED.

12,490 (1917). Fernbach. Production by fermentation of acetone and higher alcohols, particularly butyl alcohol. (109,969.) Apr. 20.

694 and 8114 (1920). Power Gas Corp., and Langwell. Fermentation of cellulose. (161,294.) Apr. 20.

16,395 (1920). Romer, and Deutsch-Koloniale Gerh- u. Farbstoff-Ges. Fermentation process. (161,870.) Apr. 27.

21,673 (1920). Stouffs. Preservation of by-products of brewing, distilling, etc., for use as animal foods. (161,885.) Apr. 27.

23,828 (1920). Warwick. Apparatus for fermentation of beer etc. (161,496.) Apr. 20.

#### XIX.—FOODS; WATER PURIFICATION; SANITATION.

##### APPLICATIONS.

Abbott, McNeill, Macredie, and Remus. Production of meat powder. 10,682. Apr. 12.

Akt.-Ges. f. Stickstoffdünger. 10,608. *See* I.

Chalas. Manufacture of a dry and soluble extract of roasted coffee. 11,046. Apr. 15. (Fr., 16.4.20.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

32,239 (1919). Forget-Me-Not, Ltd., and Hutchinson. Manufacture of alimentary products containing water-soluble vitamins. (161,238.) Apr. 20.

643 (1920). Farmachidis. Food preparation. (161,291.) Apr. 20.

1135 (1920). White (Collis Products Co.). Desiccating buttermilk etc. (161,673.) Apr. 27.

1800 (1920). Collis Products Co. Apparatus for desiccating buttermilk, sour milk, etc. (138,092.) Apr. 27.

1972 (1920). Barbet et Fils et Cie. Manufacture of grape honeys and fruit syrups. (153,548.) Apr. 27.

2695 (1920). Linden. Apparatus for treating effluent and other liquids. (161,735.) Apr. 27.

3645 (1920). Clements. Producing materials suitable for purifying air. (138,649.) Apr. 27.

21,673 (1920). Stouffs. *See* XVIII.

#### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

##### APPLICATIONS.

Alby United Carbide Factories, Ltd., and Dodd. Manufacture of salts of guanidine. 11,646. Apr. 22.

Büchler. Production of borodisalcyclic acid. 11,553. Apr. 21.

Courtaulds, Ltd., and Delph. Manufacture of carbon bisulphide. 11,771. Apr. 23.

Riedel A.-G. Soluble mercury compounds. 11,068. Apr. 15. (Ger., 16.4.20.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

2787 (1916). Weizmann and Legg. Manufacture of secondary butyl alcohol. (161,591.) Apr. 27.

12,490 (1917). Fernbach. *See* XVIII.

1140 (1920). Ellis (Soc. Chim. Usines du Rhône). Manufacture of oxaldehydes and their derivatives. (161,679.) Apr. 27.

2196 (1920). Thomson. Detoxication of the tubercle bacillus and other germs for the preparation of vaccines. (161,341.) Apr. 20.

11,691 (1920). Classen. *See* XVII.

24,192 (1920). Elektro-Osmose A.-G. Process for making specific vaccines. (150,319.) Apr. 20.

#### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

##### APPLICATIONS.

Davies. Manufacture of photographic papers. 10,949—10,951. Apr. 14.

Marks (Daylight Film Corp.). Photographic reproduction. 10,686. Apr. 11.

Martinez. Colour sensitised supports for colour photography etc., and treatment thereof. 10,773. Apr. 13.

Warneuke. Colour photography. 11,294. Apr. 19.

Wolff. Colour photography and cinematography. 11,556. Apr. 21. (Ger., 14.8.20.)

#### XXII.—ANALYSIS.

##### APPLICATIONS.

Allen and Hanburys, Ltd., and Lizius. Titration and neutralisation of chemical compounds. 11,659. Apr. 22.

Svenska Aktiebolaget Mono. Gas-analysing apparatus. 11,359. Apr. 19. (Sweden, 23.4.20.)

**I.—GENERAL; PLANT; MACHINERY.**

*Water; Heat conductivity of — between 0° and 80° C.* M. Jakob. Ann. Phys., 1920, [4], 63, 537—570. Chem. Zentr., 1921, 92, II., 689—690.

THE heat conductivity of water in C.-G.-S. units (cals. per cm. per sec. per 1° C.), between 0° and 80° C., is given correct to 1% by the equation  $\lambda = 0.001325 (1 + 0.002984t)$ .—A. R. P.

**PATENTS.**

*Liquids, pulpy masses, and the like; Process and apparatus for solidifying in fine powder —.* [Manufacture of soap powder.] Metallbank und Metallurgische Ges. A.-G. E.P. 145,048, 15.6.20. Conv., 16.4.18.

THE liquid (e.g., molten soap) is sprayed centrifugally and subjected to the action of a current of cold air which is continuously circulated through the spraying apparatus and a cooler.—B. M. V.

*Reaction towers [; Filling for —].* H. Schmidt. E.P. 147,213, 7.7.20. Conv., 11.4.19.

METALLIC strip is perforated and corrugated with corrugations of equal pitch, the strips being laid together with the tops of the corrugations touching so that a honeycomb structure is formed. —B. M. V.

*Vapours of volatile liquids; Process and apparatus for the recovery of — from their mixtures with air.* E. Barbet et Fils et Cie. E.P. 149,691, 3.3.20. Conv., 1.8.19.

AIR escaping from a storage vessel containing a volatile liquid is passed through a washing column. The column contains a suitable solvent (e.g., heavy tar oil for benzene) which is fed from a storage tank only when there is positive pressure in the storage vessel, i.e., when air is escaping. The control of the solvent feed is effected by a float in a two-compartment tank, closed at the top, and partially filled with the solvent liquid; the two compartments communicate only below the level of the liquid. One compartment (preferably the larger) contains the float and valve for supplying the solvent liquid, which flows from the other compartment to the washing column through a hand-adjusted outlet situated just above the level reached by the liquid when the float valve closes. The arrangement is made very sensitive by putting the air space above the float in communication with the air space in the storage vessel. A separate liquid seal device is provided through which air can be drawn into the storage vessel if the pressure therein falls below a predetermined limit. —B. M. V.

*Drying apparatus.* L. Eberts. E.P. 160,717, 19.8.20.

THE material to be dried is admitted into a casing within which rotates a drum heated by a fluid which is admitted and exhausted through the hollow trunnions of the drum. Stirrers attached to the drum gradually propel the material to an outlet, and additional heating surface is provided by means of tubes projecting radially from the drum, either bent to a U-shape and having both ends communicating with the drum or having their outer ends connected with longitudinal tubes which are in turn connected with spiral tubes leading to the outlet. The radial tubes may be wedge-shaped in cross-section.—B. M. V.

*Drying apparatus.* T. Izumi. U.S.P. 1,372,352, 22.3.21. Appl., 10.6.20.

A VERTICAL cylindrical drying chamber is fitted

with shelves revolving around a central cylindrical heat radiator, and means are provided for drawing air from the bottom of the chamber and causing it to escape through a passage around the chamber, air pipes within the passage serving to supply fresh air to the top of the chamber. The lowest part of the chamber communicates with a fluid pit provided with a lid.—H. H.

*Drying apparatus.* Z. Yamamoto. U.S.P. 1,372,585, 22.3.21. Appl., 28.11.19.

A DRYING apparatus comprises a casing in which is mounted a movable frame carrying a zig-zag shoot having an inlet at its top and an outlet at its bottom. Means are provided for heating the interior of the casing, and the shoot has openings to permit the heated air to enter.—H. H.

*Dryer.* W. F. Schaller, Assr. to E. and I. H. Schaller. U.S.P. 1,373,767, 5.4.21. Appl., 11.9.18.

THREE walls of the drying chamber are surrounded by a hot-air chamber below which is a fire chamber with flues for the products of combustion, and there are also provided flues for supplying fresh air to the drying chamber and for heating it, a flue extending downwards from one end of the drying chamber and formed with an opening at its lower end for mixing the incoming air, and a flue at the opposite end of the drying chamber for escape of the heated air.—H. H.

*Cooling towers.* L. R. Morshead. E.P. 160,990, 17.1.20.

JETS of water are arranged at the bottom of the tower, pointing upwards, in order to induce a more powerful current of air, especially in those parts where by natural draught there would be little or no current and to prevent water being blown out by cross winds. Falling water which would fall directly on the jets may be deflected by louvres. —B. M. V.

*Heating and cooling liquids; Apparatus for —.* C. W. Desobry. U.S.P. 1,373,953, 5.4.21. Appl., 17.3.19.

THE liquid to be cooled flows downwards in a zig-zag path over vertical inner walls of chambers which are divided into two similar sections, hinged at one side so that they can be opened and closed. Heating or cooling means may be installed between the inner and outer walls of the chambers. —B. M. V.

*Electrical precipitation [of vaporised substances from gases].* A. F. Nesbit. U.S.P. 1,371,995, 15.3.21. Appl., 10.12.20.

THE gases are passed through an apparatus containing several pairs of electrodes, each pair consisting of a discharge electrode and a tubular collecting electrode, producing between them an ionisation field which is cooled by a refrigerating jacket surrounding the collecting electrode and in which vaporised substances are deposited from the gases. Traps are provided at intervals along the collecting electrodes.—J. H. L.

*Cleaning gases; [Electrical] method of and apparatus for —.* F. R. McGee and A. F. Nesbit. U.S.P. 1,372,710, 29.3.21. Appl., 13.3.18.

THE gas is passed through a hollow electrode in which a corona discharge is maintained. A whirling motion is imparted to the column of gas, whereby impurities are impelled centrifugally to the periphery. The central core of clean gas and the peripheral layer of gas laden with impurities are passed into separate chambers.—J. S. G. T.



*Dust separator; Centrifugal* — H. Milliken, Assr. to J. B. Taylor. U.S.P. 1,372,714, 29.3.21. Appl., 20.9.19.

A CENTRIFUGAL dust separator for gases comprises a tubular envelope provided with one or more helical slits affording a free passage radially and extending around the periphery of the envelope. A structure within the envelope affords a continuous helical passage in the same general direction as the helical slit or slits.—J. S. G. T.

*Gases; Process and apparatus for removing vaporized, liquid and solid substances from — by washing.* W. Freytag. G.P. 331,321, 20.8.18.

THE gas is passed through a rotary wheel-shaped filtering apparatus containing a porous material which is automatically moistened, but not overcharged, with crude oil, tar, heavy oil or the like. When the filtering material is no longer effective it is automatically replaced by fresh. Materials such as porous lignite or peat are preferably employed so that after use they can be distilled in order to recover the oily washing liquid.—J. H. L.

*Feed-water heater, evaporator, and the like.* H. C. Davis, Assr. to American Marine Equipment Corp. U.S.P. 1,372,010, 22.3.21. Appl., 19.9.18.

A HORIZONTAL shell has an inlet and outlet for steam, inlet and outlet connexions for water, and a nest of straight horizontal tubes in communication with the water connexions, each tube being provided with a longitudinal indentation extending spirally around it.—H. H.

*Filter apparatus.* O. Congleton; J. T. Lewis, exor. U.S.P. 1,372,119, 22.3.21. Appl., 4.4.18.

FILTERING material is disposed between an upper chamber and a tapering lower chamber of a filter tank. The lower chamber has a valved outlet, and a connexion is provided from a supply tank to the lower chamber, such connexion having an upwardly inclined portion entering the tapering wall of the lower chamber. A series of such filter tanks may be provided, the valved outlets from the lower chambers leading to a common pipe, and each upper chamber having a pipe connexion to the lower chamber of the succeeding tank. The upper chamber of the final tank is provided with an outlet pipe, and the supply tank is connected with the upper chambers of the various filter tanks.—H. H.

*Filtration process.* D. P. Hynes, Assr. to Roseberry Surprise Mining Co., Ltd. U.S.P. 1,373,887, 5.4.21. Appl., 26.1.20.

A BASIC compound of zinc is added to a pulp containing solid and colloidal substances and substantially free from acid, whereby the colloidal substances are changed to a form which does not inhibit filtration.—A. de W.

*Distilling column.* G. W. Crupe, Assr. to Semet-Solvay Co. U.S.P. 1,372,649, 22.3.21. Appl., 23.10.20.

IN a distilling column having a series of superimposed pans with an external overflow passage from pan to pan, there is a vent pipe connexion from the upper part of each such passage, and the vent pipe has a discharge orifice within the column, above the opening from the pan to the passage.—H. H.

*Still.* C. D. Crane. U.S.P. 1,372,865, 29.3.21. Appl., 18.7.17.

IN a still of the type in which the main shell is divided into a lower generator compartment and an upper compartment for condensing medium, the division wall forming the condensing surface, a settling chamber is arranged external to the main

shell. A fluid supply conduit leads from the upper compartment of the still to the settling chamber, and an overflow connexion leads from the settling chamber to the lower compartment of the still.—H. H.

*Distilling and purifying apparatus for volatile liquids.* H. A. Hills. U.S.P. 1,372,880, 29.3.21. Appl., 18.8.16.

A STILL having a sloping bottom, a vapour outlet at the top, and a liquid inlet is provided with a nozzle having its discharge opening directed against the bottom. A sleeve forms an annular chamber around the nozzle, and an opening is provided at the rear of the chamber through which fluid is supplied by suction simultaneously with the exhaust from the nozzle.—H. H.

*Crucible furnace.* J. H. L. De Bats, Assr. to Lava Crucible Co. U.S.P. 1,372,676, 29.3.21. Appl., 3.3.17. Renewed 5.8.20.

A crucible furnace comprises a main chamber with a bottom, adapted to be lowered and lifted, on which is erected a crucible-receiving chamber with non-perforated heat-conducting walls of refractory material, spaced from the walls of the main chamber to provide a fire space. The bottom of the space surrounding the crucible-receiving chamber is formed as a grate.—H. H.

*Catalytic material and process of making the same.* A. A. Backhaus, Assr. to U.S. Industrial Alcohol Co. U.S.P. 1,372,736, 29.3.21. Appl., 19.12.18.

A CATALYTIC material is made by applying syrupy phosphoric acid to coke, using a diminished pressure before and during the application, then restoring the pressure and reducing the acid content to about 15%.—H. H.

*Centrifugal separation; Process for* — E. W. Beach. U.S.P. 1,373,219, 29.3.21. Appl., 31.1.21.

FOR separating a heavier solid from a lighter fluid constituent of a mixture, a centrifugal machine is used having a discharge passage near its outer wall and another discharge passage relatively close to its centre of rotation, and there is introduced into the machine a sealing and carrying fluid of a specific gravity intermediate between the specific gravities of the constituents to be separated. Sufficient of the sealing liquid is used to form a layer against the outer wall to close completely the outer discharge passage. This layer prevents the lighter constituent from passing through that passage while allowing the heavier constituent to be removed with the sealing material.—H. H.

*Centrifugally separating substances; Method and mechanism for* — L. D. Jones, Assr. to The Sharples Specialty Co. U.S.P. 1,373,743, 5.4.21. Appl., 4.9.20.

THREE substances of differing densities are separated by subjecting them to centrifugal force and controlling the distance of the contact surfaces of the stratified constituents from the centre of revolution so that either contact surface of the intermediate layer may vary substantially without substantially varying its other contact surface.—H. H.

*Emulsions; Method of resolving* — P. T. Sharples. U.S.P. 1,373,773, 5.4.21. Appl., 21.10.18.

A THREE-PHASE emulsion is centrifuged to effect separation with the separate discharge of the lightest phase. The discharge is retarded so as to hold a relatively large volume of the lightest phase in the centrifuge, and this is subjected to the highest centrifugal force which is practicable without any substantial discharge of the lightest phase with the heavier phases.—H. H.

*Centrifugal separation; Method of and machine for —.* W. H. Peck. U.S.P. 1,374,098, 5.4.21. Appl., 14.11.19.

THE mixture is stratified centrifugally, the heavier matter is discharged through ports, and fluid under pressure is caused to act through the ports against the outflow of the heavier particles and then to discharge with the lighter particles through other ports.—H. H.

*Extracting valuable components [soluble salts] from raw materials; Process of —.* J. W. Hornsey. U.S.P. 1,373,731, 5.4.21. Appl., 29.6.16.

Raw material containing the soluble salt together with other ingredients is caused to move in contact with and in a counter direction to a mother liquor saturated with other ingredients, within the range of working temperatures employed. The liquor is agitated, solid and liquid are separated, and the liquid is removed from a quiet zone remote from the agitated zone.—J. S. G. T.

*Thickener (sewage type). Automatic separator.* C. Allen. U.S.P. (A) 1,374,134, and (B) 1,374,135, 5.4.21. Appl., 11.3 and 8.6.20.

A SETTLING vessel is provided with an overflow rim and with a discharge pipe for settled material which extends nearly to the bottom. In (A) the discharge pipe is brought out through the side of the vessel near the top (siphon action) and the rate of discharge controlled by a valve actuated by a float at about the level of the settled material. In (B) the fixed discharge pipe extends vertically upwards above the vessel; the vessel itself is mounted on springs and provided with a valve which closes the discharge pipe when the vessel is light but opens when the vessel reaches a pre-determined weight, the discharge being effected by a fluid pressure pipe opening into the discharge pipe, e.g., by an air lift.—B. M. V.

*Furnace.* A. Smallwood. U.S.P. 1,373,152, 29.3.21. Appl., 17.7.20.

SEE E.P. 153,042 of 1919; J., 1920, 809 A.

*Charging apparatus for furnaces, kilns, and the like.* C. Candiot. E.P. 137,297, 29.12.19. Conv., 10.7.17.

*Pulverised coal and other material; Feeding — to furnaces [by means of air under pressure].* P. E. van Saun. E.P. 160,866, 8.10.19.

*Drying chamber.* E.P. 147,562. See XIII.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal; Development of electrical conductivity in — and its variation as a result of heating.* F. Fischer and G. Pfeleiderer. Ges. Abhandl. Kennt. Kohle, 1919, 4, 394–408. Chem. Zentr., 1921, 92, II., 521–522. (Cf. Sinkinson, J., 1920, 591 A.)

COAL, peat, lignite, and wood when carbonised all develop an appreciable electrical conductivity at the same temperature, viz., 700°–750° C. The change of conductivity as a result of heating was explored up to a temperature of 1100° C. The temperature coefficient of conductivity was greater with the moderately heated specimens than with the more highly heated specimens. The low-temperature (up to 600°) carbonisation products of the different fossil coals were all in the same condition as regards electrical conductivity. The results suggest a means for distinguishing between low and high temperature cookes, for determining their temperature of carbonisation, and for controlling the uniformity of heating in the charge of coke ovens.—H. J. H.

*Coals; Methods for the identification and valuation of —.* F. S. Sinnatt. J. Soc. Dyers and Col., 1921, 37, 108–113.

A KNOWLEDGE of the agglutinating power of a coal (J., 1920, 837) enables the consumer to compensate for the variation in caking power by varying the amount of coke mixed with the coal, or by varying the size of the particles of the coke breeze, so as to ensure efficient combustion of the coal in steam raising. The rate of combustion of the coal can be modified by the addition of water (up to 8%) prior to introduction into the grate. A further factor in efficient combustion is the average actual size of the pieces of coal.—W. P.

*Lignites; Some constituents of —.* R. Ciusa and A. Galizzi. Gazz. Chim. Ital., 1921, 51, 1, 55–60.

THE xyloid lignite of Fognano, near Montepulciano, contains a hydrocarbon,  $C_{15}H_{32}$ , m.pt. 61°–62° C., b.pt. 314°–316° C., or 208°–210° C. at 23 mm. pressure, which represents a new mineral species and is named simonellite; and also three unsaturated hydrocarbons:  $C_{15}H_{28}$ , b.pt. 222°–228° C. at 25 mm.;  $C_{15}H_{26}$ , b.pt. 240°–244° C. at 25 mm., and  $C_{15}H_{24}$ , b.pt. 253° C. at 25 mm. A lignite deposit near Terni contains a hydrocarbon,  $C_{15}H_{24}$ , m.pt. 74°–75° C., which is possibly identical with hartite. The material known commercially as Forli lignite has more the character of peat and contains lignoceric acid. (Cf. J.C.S., May.)—T. H. P.

*Tar; Increase in yield and quality of — by cooling the crude gas between the retort and hydraulic main.* Binder. Gas- u. Wasserfach, 1921, 64, 129–130.

IN the experimental apparatus used the gas was sprayed with the ammoniacal liquor in the horizontal oftake to the hydraulic main. It was then passed through a tar separator, a cleaning tower packed with cotton wool, and a tower packed with ferric oxide. The water from the main was led off into two tanks, from the second of which it ran to waste quite clear and from the first of which the tar could be drained off and any scum removed. With the adoption of spraying, 60.3% of the tar was recovered in the hydraulic main, whilst without spraying the quantity recovered in the main was 19.3% of the total. From one ton of coal there was obtained by spraying 41.1 kg. of tar, whilst without spraying there was obtained only 32.8 kg. of tar and 4.6 kg. of pitch. It is claimed that such spraying prevents the formation of pitch in the mains, and the quality of the tar is improved.—A. G.

*Gases; Behaviour of explosive mixtures of — at low pressures.* A. Stavenhagen and E. Schuchard. Z. angew. Chem., 1921, 34, 114–115. (Cf. J., 1920, 812 A.)

THE critical pressure depends on the diameter of the explosion tube; as the diameter increases the critical pressure decreases. This is due to the greater cooling effect of the walls of the larger tubes. When a spark is passed continuously through a mixture of oxygen and hydrogen maintained below the critical pressure at which explosion occurs, the gases combine slowly and without producing light.—W. P. S.

*Ignition of gaseous mixtures by induction coil sparks; Phenomena of the —.* J. D. Morgan and R. V. Wheeler. Chem. Soc. Trans., 1921, 119, 239–251.

MEASUREMENTS of the minimum currents ("igniting currents") required in the primary to produce sparks in the secondary of an induction coil at a gap of constant width capable of igniting mixtures in various proportions of methane and air, indicated that the igniting current increases as the percentage of methane in the mixture increases or decreases

on either side of a critical value of 8.2–8.4%. The voltage of the sparks bears a similar relation to the methane percentage, as does also the width of the igniting gap with a constant current of 1 amp. in the primary, so that the energy represented by a given igniting gap bears the same relationship to the mixture ignited as does the energy corresponding to the igniting current for that mixture. As, however, it was shown by thermal measurements made on the sparks that the total energy of the sparks bore a very different relationship to the percentage of methane in the mixture ignited, whilst the voltage of the sparks produced at given igniting gaps bore a relationship similar to that found in the ignition experiments, it is concluded that the igniting power of the induction coil sparks is to be ascribed to the energy of their "capacity components" only ( $J = \frac{1}{2} CV^2$ ), and not to their total energy. The assumption involved in this deduction, that ignition is essentially a thermal process involving the heating of a sufficient volume of the mixture to a sufficient temperature for a sufficient length of time, is shown to be justifiable.

—G. F. M.

*Air-benzine mixtures; Determination of higher and lower limits of explosion of —.* W. Reinders. Chem. Weekblad, 1921, 18, 157–159.

For determining the higher and lower limits of explosibility of air saturated with benzine vapour, an apparatus is used consisting of a wide glass tube, surrounded by a jacket containing water or a freezing-mixture, with two platinum wires fused in the top, and with its upper end terminating in a narrow tube with funnel and tap. The tube is connected with a levelling tube. The apparatus is first filled with water and a layer of benzine and a few c.c. of air are then introduced. After bringing the mixture to any desired temperature, the explosibility of the air-vapour mixture is tested by passing a spark through it. Results obtained with many benzine fractions are tabulated. With the light benzine fractions used in motor-cars air saturated with the vapour at temperatures above 0° C. is not explosive; on the other hand, the heavier fractions (b.p. 100°–200° C.; sp. gr. 0.74–0.75) may constitute a risk, and their displacement from storage reservoirs in garages should be effected by means of inert gases, such as nitrogen or carbon dioxide.

—W. J. W.

*Paraffin wax; After-treatment of solution obtained in pressure oxidation of —.* F. Fischer and W. Schneider. Ges. Abhandl. Kennt. Kohle, 1919, 4, 94–100. Chem. Zentr., 1921, 92, II., 522–523.

WHEN preparing soaps by the pressure oxidation of paraffin wax (cf. Schneider, J., 1921, 290A) the soda solution employed should be neutralised as completely as possible. The soaps can be separated by direct evaporation of the solution, or, better, by removing the supernatant layer of paraffin and allowing to stand over-night, when the salts of the fatty acids separate in the solid state. Brown soaps may be bleached by sodium hypochlorite or by further treatment in the autoclave at 160° C. Still more effective is treatment of the warm solution with acid or with carbon monoxide under pressure at 180°–200° C. The carbon monoxide, behaving as formic anhydride, liberates free fatty acids with the production of sodium formate. This may be converted into formic or oxalic acid or calcined to regenerate the sodium carbonate originally used. The fatty acids precipitated may be purified by distillation either with superheated steam or *in vacuo*. Neutral substances remaining are removed by distillation under diminished pressure at 250° C., followed by extraction with benzene in the cold. In the preparation potash or mixtures of potash and

soda may replace sodium carbonate to give correspondingly different types of soap. All the soaps lather well.—H. J. H.

*Colorado shale oils; Investigation of —.* A. J. Franks. Chem. and Met. Eng., 1921, 24, 561–564.

SHALE oils from the low-temperature carbonisation of shales from various strata were distilled, and each 10% (by vol.) of the distillate was examined separately. Curves plotted to show the relation between temperature and percentage distilled show a similarity in character of the oils; a sharp bend occurs at 350°–390° C., representing the temperatures between which cracking occurs, although this takes place slightly even at 300° C. Curves obtained with composite samples from the secondary and primary condensers indicate that it is not possible to condense the oil vapours fractionally with success. Specific gravity curves confirm the similar general character of the oils. Saturation (percentage of volume not attacked by sulphuric acid) decreases with increase in boiling point and specific gravity, up to the point where cracking begins, and it is remarkable that cracking of unsaturated oils in the heavy fractions produces lighter oils of higher saturation. This phenomenon is so far without adequate explanation.—W. J. W.

*Decomposition of hydrocarbons.* Kohlschütter and Frumkin. See III.

*Gas from strawboard factory waste waters.* Hermann. See V.

#### PATENTS.

*Peat; Process for dehydrating —.* E. Laaser and C. Birk. G.P. 331,692, 28.12.19.

THE peat pulp is heated under pressure which is then released. Evolution of moist vapours occurs and the vapours are compressed and used for the drying of the peat pulp. As a result of the compression the vapours acquire a temperature somewhat higher than that of the liquid in the peat pulp, and this temperature difference is sufficient for evaporation of the liquid.—H. J. H.

*Coke ovens.* E. Hurez. E.P. 153,272, 12.3.20. Conv., 29.10.19.

A REGENERATIVE coke oven is adapted to be heated either by rich gas from the oven itself or by producer gas or blast-furnace gas. Two adjacent superposed sub-flues establish communication between the heating flues of the oven and two series of regenerating chambers, the arrangement being such that when the oven is heated with poor gas all the even chambers serve alternately for the passage of the waste gases and for the preheating of the air, and the odd chambers serve alternately for the passage of the waste gases and for the heating of the poor gas. When rich gas from the oven is used, the air necessary for combustion is heated by the entire series of regenerating chambers.

—A. G.

*Coke ovens.* Foundation Oven Corp. E.P. (A) 160,625 and (B) 160,626, 29.1.20, and (C) 160,628, 30.1.20.

(A) THE regenerator, which is located in the sole of the oven, consists of an air passage of narrow rectangular cross-section returning parallel to itself once or several times along the whole length of the sole, and having one of its wide faces in close proximity to a waste gas passage for the whole or part of its length. One or several superposed waste gas passages are in close proximity to, and surrounded laterally by, the air passages, which pass back and forth in the same horizontal plane as the waste gas passages. The flues in

adjacent rows are so arranged that the combustion flues in one row are opposite the waste gas flues in the adjacent rows, so as to distribute equally the heat throughout the oven. Two waste gas flues correspond to each combustion flue, one of the former being placed on either side of the latter. (b) A regenerator and gas preheating duct system are provided for each row of flues. The gas preheating ducts can be cut out and air and hot waste gases can be conducted alternately through a common horizontal flue in the sole of each coking chamber, which flue communicates with alternate flues in the two heating walls of the coking chamber. Two gas ducts under each heating wall are connected with alternate combustion flues in each wall. Each regenerator is divided longitudinally into two superposed portions and communicates with the common air or hot gas flue at the outer end of the oven only. The gas admission and discharge ends of the preheating ducts are exposed in front of the oven and are adapted to be easily connected respectively with a gas main and with a gas duct leading to alternate combustion flues or to be closed altogether. A three-way valve is provided outside the oven which can open communication of the discharge end of the gas preheating system with either of the two gas ducts leading to the combustion flues of each heating wall. The combustion flues are staggered, so that the vertical combustion flues in one heating wall are opposite the vertical hot gas flues in the adjacent heating walls. Each pair of combustion flues is provided with a separately adjustable valve by which the quantity of air can be adjusted separately at successive points along the line of the combustion flues so as to control the distribution of heat along this line. The air from the horizontal flue can be admitted to each pair of combustion flues independently. (c) The coke oven has combustion flues, gas ducts, and a preheating passage for the gas, the outer ends of the gas ducts comprising a box or casing providing two chambers in communication with the respective gas ducts and having ports opening communication between the respective gas ducts and the preheating passage. The ports are provided with valves, and a single device on the outside of the oven is provided for operating the valves alternately.—A. G.

*Gas; Method of manufacturing* — W. D. Wilcox. U.S.P. 1,372,731, 29.3.21. Appl., 1.4.18.

THE plant comprises two vertical cross-connected retorts disposed above and discharging into two corresponding generators. One of the generators is blown with air and the blast gases are conveyed through a valve into heating flues adjacent to the retorts. A partial vacuum is concurrently created at an outlet in the lower portion of the second generator sufficient to draw the gases and vapours distilling off in the retorts through the preheated material in the second generator and through the outlet. The second generator is then subjected to an air blast and the process repeated, using alternate generators and retorts. A maximum pressure is maintained within the plant at a point in the lower portion of the retort above the generator in blast.—A. G.

*Gas producer.* J. C. Miller and A. Isles, Assrs. to The W. R. Miller Co. U.S.P. 1,373,443, 5.4.21. Appl., 14.6.19.

THE cylindrical body and ash-pan of the producer are revolved at the same rate and in the same direction. The body carries a grate which is inclined inwards and surrounded by a stationary bustle pipe, water troughs sealing the joint between the bustle pipe and the body portion. The blast devices extend through the bustle pipe.—A. G.

*Gas; Method of producing* — L. W. Bates. U.S.P. 1,373,704, 5.4.21. Appl., 19.2.20.

A MIXTURE of a liquid hydrocarbon and a pulverised solid fuel is injected into a mixture of air and steam in a generator heated to a temperature sufficient to transform the particles of solid carbonaceous substance and the liquid hydrocarbon into gas. The ash is removed from the gas, which is then washed with a liquid hydrocarbon to remove tar and carbon.—A. G.

*Chamber oven plant.* H. Brücker. G.P. 330,347, 17.4.19. Addn. to 315,099 (J., 1920, 478 A).

THE producers are first heated with coke and then charged with coal which is carbonised by heat from the coke below, which after some degree of cooling is withdrawn. The producers thus behave as gas retorts—a convenience in small installations.

—H. J. H.

*Distillation of coal; Apparatus for* — A. Newerth. G.P. 331,508, 24.8.12.

A CENTRAL vertical retort is provided with two inclined heating chambers, one at each side, containing coal or coke, used as fuel. The free spaces of the side chambers and of the central retort are in communication and one side chamber is connected with the top of the retort, the other with the bottom. Producer gas developed by a blast in one side chamber is passed from the top of the inner retort to the bottom and thence into the hot coal in the other side chamber. The process can be reversed so as to maintain a continuous distillation.

—H. J. H.

*Vertical retort for continuous carbonisation of coal.* Rombacher Hüttenwerke, and J. I. Bronn. G.P. 332,102, 14.7.18.

MOST coking coals, when being carbonised, have completed the stage of melting, caking, and swelling by about one-quarter to one-third of the time for complete coking. Afterwards no further change in dimensions occurs, and if an enlargement of the sectional area of the retort is made at the point where this stage is reached, the hanging up of the charge can be prevented.—H. J. H.

*Gas-making furnace.* B. Ludwig. G.P. 332,103, 31.3.18.

THE furnace is provided with superposed heating flues in which gas and air are led separately, but the flues are perforated so as to permit a gradual admixture and combustion and divided into two groups in which the heating gases travel in opposite directions. Gradual combustion and even heating of the furnace are achieved.—H. J. H.

*Fuels; Treatment of dusty, bituminous — for recovery of nitrogenous by-products by carbonisation.* "Gafag," Gasfeuerungs-ges. Wentzel und Co. G.P. 332,507, 5.8.19.

THE fuels are "briquetted" by means of sodium silicate, whereby the passage of gases through the charge during carbonisation is facilitated.

—W. J. W.

*Low-temperature tar; Method for producing tar of aliphatic compounds or — [in gas producers].* G. A. Pestalozzi. E.P. 145,408, 9.6.20. Conv., 20.6.19.

IMMEDIATELY above the fuel bed in a gas producer a cooling zone is established, the temperature of which is kept below 400° C. by injecting water, steam, gases or the like, whereby detrimental decomposition of the gases and vapours produced in the distilling zone of the generator is avoided.

—A. G.

*Mixed gas from the distillation of coal and of wood, peat, or lignite; Production of —.* C. O. Rasmussen. G.P. 332,057, 17.11.18.

THE gases are not mixed until the tars have been separated and then the mixture goes through the customary purification processes. Whereas in coal gas tars basic compounds predominate, in tars from wood, peat, etc. acid constituents are in excess. If the crude gases are mixed stoppages occur readily, while the tars are commercially more valuable when separate than when mixed.—H. J. H.

*Gas; Production of lighting and heating — from bituminous shale.* E. von der Burchard and A. Rau. G.P. 332,155, 22.5.19.

CRUDE gas obtained in the distillation of shale is passed through a layer of red hot carbonaceous material, e.g., coke or charcoal, contained either in the retort or in a separate "cracking" chamber. Oils are thereby cracked to form permanent gas, and carbon dioxide from the shale is reduced to carbon monoxide. A comparatively high-grade gas (310—400 B.Th.U. per cub. ft.) is produced.—H. J. H.

*Blast-furnace and generator gases; Purification of —.* N. Klärting. E.P. 153,263, 28.10.20. Conv., 31.10.19.

THE crude gas is passed through granular material, which moves continuously downward through an enclosed chamber, and the dust is removed from the material by passing a branch stream of purified gas through the material in an auxiliary chamber, into which the material is discharged by a shaking screen in the lower part of the main chamber.—A. G.

*Hydrogen sulphide [in producer and coke-oven gases]; Oxidation of —.* W. Schumacher. G.P. 331,287, 6.3.17.

THE hydrogen sulphide in producer gas, coke-oven gas, and the like is oxidised to sulphur and oxides of sulphur by passing the gas mixture, together with the requisite amount of air, over charcoal which is heated to a temperature below that at which the gas ignites. The process can be carried out at 100° C.; the charcoal remains unchanged and the sulphur deposited on it can be recovered afterwards without difficulty.—J. H. L.

*Iron oxide solutions; Preparation of stable, alkaline — for gas purification.* Badische Anilin- und Soda-Fabr. G.P. 333,755, 3.4.18.

THE addition of a small quantity of a disinfectant, e.g., phenol, cresol, naphthol, or crude tar, to the alkaline solution prevents decomposition by bacteria of the organic compounds, such as tartaric acid or oxalic acid, added to keep the iron compounds in solution.—L. A. C.

*[Alcohol] distillation process.* E. W. Stevens, Assr. to Chemical Fuel Co. U.S.P. 1,372,465, 22.3.21. Appl., 9.3.20.

COMMERCIAL aqueous alcohol is mixed with a hydrocarbon oil and fuel oil and the mixture is redistilled to obtain a fraction consisting mainly of alcohol and substantially free from water, for use in making composite motor-fuels.—A. R. P.

*Shale and the like material; Method of and apparatus for treating —.* L. B. Ard. U.S.P. 1,373,698—9, 5.4.21. Appl., 3 and 4.2.21.

THE shale is conveyed to the bottom of a cylindrical retort containing molten metal by a helical vertical conveyor, concentric with a helical vane surrounding the conveyor which retards the upward flow of the shale, so that the whole of the volatile matter is evolved during the upward movement of the shale through the molten metal.—A. G.

*Oil-shale retort.* F. Jones. U.S.P. 1,373,890, 5.4.21. Appl., 29.5.18.

A CONTINUOUS retort for the destructive distillation of oil-sands and oil-shale comprises a series of superposed intercommunicating annular hearths, combustion or heating chambers interposed between the hearths, the floors of the latter constituting the roofs of the combustion chambers, a central hollow shaft with hollow arms and rabbles for advancing the material from one hearth to another, and means for supplying steam to the shaft and arms, the latter being perforated to allow steam to be directed on to the material under treatment.—A. de W.

*Petroleum; Extraction of — from oil-bearing sands and rocks.* W. Horwitz. G.P. 332,100, 1.4.19.

THE material is heated in a suitable vessel by pressure water or live steam, which is introduced tangentially or at an angle to the sides for preference. The mixture of oil-sand and hot water passes through the vessel without losing pressure. Substances are introduced either continuously or intermittently to facilitate extraction, e.g., benzene, toluene, common salt, sodium sulphate, etc.—H. J. H.

*Hydrocarbons; Apparatus for cracking —.* J. W. Coast, jun., Assr. to The Process Co. U.S.P. 1,372,937, 29.3.21. Appl., 22.5.17.

A HORIZONTAL cylindrical cracking still is provided with rows of scrapers in staggered relation attached to an oscillating frame provided with journals at the horizontal axis of the still. All the scrapers slide in the frame and fall by gravity, so as to be continually in contact with the still bottom for the purpose of removing coke deposits.—L. A. C.

*Petroleum products; Method of distilling —.* C. Kroll. U.S.P. 1,373,251, 29.3.21. Appl., 11.4.18.

THE vapours obtained by distilling the hydrocarbons percolate through a body of absorbent material contained in a chamber insulated to prevent loss of heat. The vapours of the high-boiling hydrocarbons are extracted in the chamber, and the remaining vapours are condensed.—L. A. C.

*Hydrocarbon oils; Treating —.* D. T. Day. U.S.P. 1,373,391, 29.3.21. Appl., 30.4.18.

A MIXTURE of high-boiling hydrocarbon oils and petroleum residues having a higher cracking point than the oils is subjected to heat treatment above 600° F. (315° C.), but below the temperature necessary to crack the residues. Low-boiling hydrocarbons are separated from the product, and the residues are added to a further quantity of the high-boiling oil.—L. A. C.

*Gasoline from crude oil; Process for the production of —.* Process for the production of hydrocarbons of low boiling-points from hydrocarbons of high boiling-points. P. Danckwardt. U.S.P. (a) 1,373,653 and (a) 1,373,654, 5.4.21. Appl., 27.8.17.

(A) MATERIAL containing high-boiling hydrocarbons is caused to react with a heated mixture of carbon, a halide, and a metal, and fractions of low and high boiling points are condensed separately from the vapours. (B) Air and steam are passed through an ignited mixture of carbon, nickel, and nickel chloride, hydrocarbon oil is added to the mixture, and the vapour generated is passed through a mixture of oil with carbon, nickel chloride, and nickel. That portion of the vapour which boils at or below 150° C. is condensed separately from portions boiling at higher temperatures.—L. A. C.

*Catalysis; Process of* — E. W. Stevens, Assr. to Chemical Fuel Co. of America. U.S.P. 1,374,119, 5.4.21. Appl., 18.3.20.

IN the catalysis of gaseous carbon compounds by means of hot metallic catalysts which are used under conditions whereby carbon would be deposited, the surface of the catalyst is sparked during the process.—A. J. H.

*Hydrocarbons such as petroleum spirit; Production of light* — Allgem. Ges. für Chem. Ind. m.b.H. G.P. 333,168, 5.12.17.

THE oils obtained by distilling petroleum or lignite tar are purified by treatment with liquid sulphur dioxide, and are then distilled with aluminium chloride, yielding 70% or more of light petroleum spirit.—L. A. C.

*Petroleum oils; Recovery of — from the acid resin obtained in refining petroleum*. Harburger Chem. Werke Schön und Co. G.P. 333,169, 11.12.18.

THE acid resin, after removal of free acid, is dried and agitated at 120°–125° C. with concentrated sulphuric acid. The product on distillation yields a moderately high boiling oil which can be employed for lighting or heating, as a lubricant, or for other purposes. The residual resin, which has a higher m.p. than the original material, is suitable for use as an insulator.—L. A. C.

*Still for cracking or distilling hydrocarbons, tar, or the like*. A. Sommer. G.P. 333,216, 21.10.15.

A METAL still is provided with a close fitting, thin-walled metallic lining which can be replaced when encrusted with carbon deposits.—L. A. C.

*Lubricating oil; Production of — from wood tar*. O. de F. Skibsted. G.P. 330,276, 27.6.19.

THE fractions boiling above 250° C. are neutralised with powdered lime, oxidised by a current of air, and then distilled. The treatment can be carried out in the tar still and during the distillation, which is interrupted for the purpose.—H. J. H.

*Lubricants; Production of — from tar distillates*. Verkaufsvereinigung für Teerzeugnisse G.m.b.H., and F. Schreiber. G.P. 330,970, 24.6.19.

THE heated tar oil is treated with sulphur in presence or absence of air. Anthracene and phenanthrene are thereby converted into soft, pitchy substances, which increase the lubricating properties of the oil. Lubricants for axles, ropes, and briquette-rolls can be prepared in a similar manner from the crude anthracene press-cake alone or with addition of oils.—H. J. H.

*Asphalt substitute; Production of* — Harburger Chem. Werke Schön & Co., and W. Daitz. G.P. 330,650, 6.7.18.

SORT pitch is heated and stirred for a long time with a paste made by incorporating with water mineral matter such as magnesia, alumina, silicates of magnesium and aluminium, or precipitated iron oxide. A temperature of 110°–115° C. is maintained, at which the water is evaporated and a product resembling Trinidad asphalt obtained.—H. J. H.

*Montan wax; Process for saponifying — with simultaneous bleaching of the soap produced*. F. Fischer. G.P. 334,155, 18.3.19.

CRUDE or purified montan wax is heated below 160° C. and under increased pressure with aqueous solutions of bases or other compounds which combine with fatty acids, e.g., sodium or potassium carbonate, in the presence of oxygen. By acidification of the solution a clear, hard, resinous product is obtained.—L. A. C.

*Tar-extractor and scrubber for gas*. J. Wella. U.S.P. 1,373,280, 29.3.21. Appl., 13.9.19.

SEE E.P. 153,109 of 1919; J., 1920, 813 A.

*Hydrocarbons; Process of converting liquid — into hydrocarbons of lower boiling point*. W. M. McComb. E.P. 160,907, 24.12.19.

SEE U.S.P. 1,337,144 of 1920; J., 1920, 396 A.

See also pages (A) 346, *Hydrogen sulphide from gases* (G.P. 299,163 and 331,322). 356, *Emulsions* (U.S.P. 1,373,661). 359, *Tanning material* (G.P. 333,403). 367, *Waste water from gas-works* (G.P. 333,703). 369, *Fatty acids and aldehydes* (G.P. 332,478).

## IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

"Kelpchar," a new decolorising carbon. J. W. Turrentine, P. S. Shoaff, and G. C. Spencer. Science, 1919, 50, 507.

SUPPLEMENTING the work of Zerban (J., 1919, 527 A, 873 A) it was shown that a decolorising carbon of high activity could be produced in large quantity, the method of heating being of importance. Carbonising in one stage failed to yield a product of uniform quality, but by carbonising in two stages consistently satisfactory results were obtained. After leaving the retorts, the porous char was extracted with hot water to remove potassium chloride and iodide, the residue heated with dilute hydrochloric acid, washed with water to neutrality, and dried. The carbon obtained in this way compares favourably with "Norit."—J. P. O.

### PATENTS.

*Retort [for wood distillation]*. H. M. Barr. U.S.P. 1,373,702, 5.4.21. Appl., 24.5.20.

A METALLIC shell lined with asbestos is divided by a false bottom of reticulated material into an upper retort chamber and a lower heating chamber. A pair of flues extends along the heating chamber for heating the retort, each flue including two stationary sections, one seated in one wall of the shell and leading to the source of heat, and the other seated in the opposite wall and leading to the stack. A "floating" flue section is suspended within the heating chamber between the stationary sections and is spaced from the walls of the heating chamber. The "floating" section consists of a split asbestos sleeve having a central convex portion, a split metallic sleeve having a corresponding convex portion, and clamping bands for securing the ends of the sleeve to the respective flue sections.—A. G.

*Wood and other vegetable products; Recovery of volatile compounds from* — E. Heuser. G.P. 332,298, 8.4.19.

THE raw material is heated with inorganic acids at normal or elevated pressure, with or without the introduction of steam, and is afterwards subjected to dry distillation. A good yield of volatile organic compounds results without any reduction in the yield of tar or gases.—W. J. W.

*Low-temperature tar; Apparatus for production of* — G. de Grahl. G.P. 332,109, 19.3.19.

THE raw material (coal, peat, or similar substances) is continuously forced or drawn through tubes enclosed in spiral heating flues, the tubes being arranged singly or in series. The products are delivered into a chamber, where separation of the gas from the solid residue occurs, so that the time of contact of gas and solid is reduced to a minimum.—H. J. H.



### III.—TAR AND TAR PRODUCTS.

**Hydrocarbons; Decomposition of — by canal rays.**  
V. Kohlschütter and A. Frumkin. Ber., 1921, 54, 587—594.

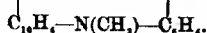
THE vapours of naphthalene, benzene, diphenyl, anthracene, phenanthrene, fluorene, decane, and petroleum have been subjected to the action of canal rays in a vessel so designed that the vapour can be passed at a desired pressure between an anode of aluminium wire and a specially constructed cylindrical cathode. In every case the latter becomes more or less rapidly coated with a deposit which ultimately is of sufficient thickness to impede the passage of the current. The properties of the deposit depend on the potential of the cathode and not on the mode of union of the carbon in the decomposed product. With low potentials the deposits are transparent and coherent, and evolve large quantities of gas and tar when heated; at higher potentials they consist of practically pure carbon, which closely resembles anthracite in appearance. Graphite does not appear to be formed. (Cf. J.C.S., June.)—H. W.

**Phenols; Pressure oxidation of —.** F. Fischer and H. Schrader. Ges. Abhandl. Kennt. Kohle, 1919, 4, 293—309. Chem. Zentr., 1921, 92, II., 606—607.

THE oxidation of phenols by air under 45 atm. pressure was examined to see if resinous substances were obtainable, with the ultimate object of producing substances similar to bakelite from the phenols of low-temperature tar without the use of formaldehyde. The products obtained were partly insoluble condensation products and partly degradation products. Under suitable conditions fairly clear resinous substances were obtained, the colour being dependent on the reaction and concentration of the original mixture. Phenol at 100° C. in presence of a 2.5 N soda solution yielded a considerable quantity of brown resinous product, but less at 150°—200° C. The three isomeric cresols were oxidised in presence of soda solution and *o*-cresol in presence of an excess of sodium hydroxide. Colouring matters and degradation products were obtained, but little resinous matter. The colouring matters were apparently fuchsine derivatives resulting from condensation of cresol and aldehydes. In presence of N/1 H<sub>2</sub>SO<sub>4</sub> above 150° C. *o*-cresol was extensively oxidised, yielding solid products. At 150° C. the products were a hard, lustrous, orange-coloured resin, soluble in benzene and alcohol, and also a brown substance insoluble in benzene, and small quantities of colouring matter.—H. J. H.

**Naphthols and naphthylamines; Reaction of — with bisulphite.** P. Friedländer. Ber., 1921, 54, 620—624.

THE reactivity of naphthols with bisulphite has been considered by Bucherer to be due to the intermediate formation of highly reactive naphthyl sulphites, but this view is not in harmony with the established properties of these substances. It is simpler to assume that the naphthols react in the tautomeric ketonic form, and that the products are normal ketone bisulphite additive compounds. Evidence in favour of this view is found in the observation that hydroxynaphthoic acid and bisulphite react with *α*-methylphenylhydrazine in exactly the same manner as with phenylhydrazine and yield N-methylnaphthophenocarbazole,



This reaction is not possible if Bucherer's view be accepted. (Cf. J.C.S., June.)—H. W.

**Toluenesulphonyl chlorides; Melting point of mixtures of *o*- and *p*-** L. Harding. Chem. Soc. Trans., 1921, 119, 260—262.

THE melting points of mixtures of pure *o*- and *p*-toluenesulphonyl chlorides in various proportions were determined and the melting point curve obtained. Following are some of the figures recorded: 0.0 % of *para*-isomeride, m.p. 10.17° C.; 10.0 %, 5.8°; 17.5 % (eutectic), 1.6°; 20.0 %, 7.9°; 50.2 %, 39.8°; 78.2 %, 57.3°; and 100.0 % of *para*-isomeride, 67.2° C. The pure *o*-toluenesulphonyl chloride was prepared by fractional precipitation of the *o*-sulphonamide from its sodium salt, hydrolysis of this substance to sodium *o*-toluenesulphonate, and treatment of the latter with phosphorus pentachloride. It had the following constants: b.p. 126° C. at 21 mm., sp. gr. 1.3383 at 20°/4° C., m.p. 10.17° C.,  $n_D^{20}$  = 1.5575.—G. F. M.

**Dinitroanthraquinones.** M. Battegay and J. Claudin. Bull. Soc. Ind. Mulhouse, 1920, 86, 623—631.

WHEN the crude product of the dinitration of anthraquinone is reduced the whole of the 1.5-, almost the whole of the 1.8-, and a large proportion of the 2.6-diaminoanthraquinone can be separated. The residue of mixed diaminoanthraquinones has been diazotised, converted into the corresponding dihydroxyanthraquinones, and separated into 2.6-, 1.8-, 1.6-, 1.7-, and 2.7-dihydroxyanthraquinones. Based on these results, it is calculated that the dinitration of anthraquinone yields 37% of 1.5-, 37% of 1.8-, 4.2% of 1.7-, 3.6% of 1.6-, 6% of 2.6-, and 4% of 2.7-dinitroanthraquinone. Dhar (J., 1920, 635 A) claimed that 1.2-, 1.3-, 2.6-, and 1.5-dinitroanthraquinones are formed by the dinitration of anthraquinone, and identified these products by conversion into the corresponding dihydroxyanthraquinones. Repetition of his experiments has led to the isolation of 1.5- and 1.8-dihydroxyanthraquinones, and has shown that the products regarded by Dhar as 1.2- and 1.3-dibromoanthraquinones cannot be homonuclear dibromoanthraquinones. (Cf. J.C.S., June.)—F. M. R.

**Dibromoanthraquinones.** M. Battegay and J. Claudin. Bull. Soc. Ind. Mulhouse, 1920, 86, 632—635.

THERE are ten possible dibromoanthraquinones, and that used by Graebe and Liebermann as the intermediate product in their classical synthesis of alizarin might be assumed to be the 1.2-derivative, but a study of various dibromoanthraquinones has shown that that is not the case. The dihydroxyanthraquinone of Graebe and Liebermann has been stated to be formed by the direct bromination of anthraquinone at 100° C. or by the oxidation of tetrahydroanthracene, but the two products are not identical. In the former case the product is 2.7-dihydroxyanthraquinone, yellow spangles, m.p. 252° C., whilst in the latter case, after many crystallisations from benzene and toluene, 2.3-dihydroxyanthraquinone, yellow needles, m.p. 283° C., is obtained. When 1.4-nitroanthraquinone is reduced, the bromine is eliminated, and  $\alpha$ -aminoanthraquinone is formed. (Cf. J.C.S., June.)—F. M. R.

#### PATENTS.

**Tar oils; Production of pure — from the crude oils.** M. Melamid and L. Grötzinger. G.P. 332,112, 5.4.17. Addn. to 264,811 (E.P. 9856 of 1912; J., 1913, 820).

CRUDE tar oils, or fractions thereof, are treated with phosphoric acid or similar compounds in the cold, followed, if necessary, by distillation. Unsaturated hydrocarbons are polymerised and separate with the phosphoric acid as resinous masses.—H. J. H.



*Phthalic anhydride; Manufacture of* — A. Wohl. E.P. 145,071, 16.6.20. Conv., 28.6.16.

PHTHALIC anhydride is obtained by passing a mixture of naphthalene vapour with air or oxygen over a suitable catalyst maintained below 500° C. The gas mixture may conveniently be obtained by passing air through or over naphthalene at 100°–120° C., and a suitable catalyst is prepared by soaking pumice (18 pts.) in a solution containing vanadic acid (2 pts.), and drying. The best results are obtained when the contact mass is maintained at 320°–330° C.—G. F. M.

*Diphenyl oxide; Process of forming* — J. M. Johlin, Assr. to Semet-Solvay Co. U.S.P. 1,372,434, 22.3.21. Appl., 24.7.19.

A MIXTURE of sodium benzenesulphonate and sodium hydroxide in such proportions that a portion of the sodium benzenesulphonate remains uncombined is heated to produce sodium phenoxide, and the heating is continued at such a temperature that the sodium phenoxide combines with the unchanged sodium benzenesulphonate to yield diphenyl oxide. —L. A. C.

*Nitrotetrahydronaphthalenes; Preparation of reduction products of* — Preparation of polycyclic, hydroaromatic hydrocarbons (octahydroanthracene and octahydrophenanthrene). Tetralin Ges.m.b.H. G.P. (A) 333,157, 17.3.16, and (B) 333,158, 15.3.19.

(A) THE nitro derivatives of tetrahydronaphthalene, or its homologues, yield on reduction with different reducing agents not only the corresponding amines, but also intermediate reduction products, such as hydroxylamines, which by intramolecular conversion yield derivatives of *p*-aminophenol, azoxy-, azo-, and hydrazo-compounds, the last of which undergo the benzidine conversion. A mixture of 1- and 2-nitrotetrahydronaphthalene on reduction, e.g., with tin or stannous chloride and hydrochloric acid, yields a mixture of the corresponding amines, which can be separated by fractional crystallisation of the hydrochlorides or the acetyl compounds; 1- and 2-acetaminotetrahydronaphthalenes melt respectively at 156°–158° C. and 105°–106° C.; 1-amino-tetrahydronaphthalene is a colourless liquid when freshly distilled, h.p. 146° C. (12 mm.), and 2-amino-tetrahydronaphthalene forms colourless crystals, m.p. 38.5°–39.5° C., b.p. 146°–147° C. (12 mm.). Pure 1-nitrotetrahydronaphthalene, prepared by partial reduction of 6 mols. of a mixture of the 1- and 2-nitro compounds with 10 mols. of hydrogen and subsequent shaking with acid to dissolve the amines, on reduction with sufficient zinc dust and alcoholic sodium hydroxide to give 4 atoms of hydrogen, yields 1-azotetrahydronaphthalene, which forms ruby red prisms from hot acetic acid, m.p. 175°–177° C., and can be converted by further reduction with zinc dust and sodium hydroxide to 1-hydrazotetrahydronaphthalene, m.p. 180°–182° C. This compound is readily oxidised to the azo compound, and on treatment with mineral acids gives 4,4'-diamino-1,1'-octahydrodinaphthyl. 4-Nitro-2-aminotetrahydronaphthalene, m.p. 94°–96° C., is prepared by nitrating 2-acetaminotetrahydronaphthalene, by reducing a solution of 1,3-dinitrotetrahydronaphthalene in boiling alcohol and ethyl acetate with sodium hydrosulphide, or by partial catalytic reduction of the dinitro compound; complete catalytic reduction of the dinitro compound with hydrogen at 125° C. under pressure yields 1,3-diaminotetrahydronaphthalene, h.p. 201°–203° C. (13 mm.) and m.p. 72°–74° C. A solution of 3-nitro-2-aminotetrahydronaphthalene in decahydronaphthalene on treatment with hydrogen at 150° C. under pressure, in the presence of nickel, yields 2,3-diaminotetrahydronaphthalene, m.p. 135°

–136° C., which on boiling with acetic acid forms an iminazole,  $C_{12}H_{10} \begin{smallmatrix} \diagup N \\ \diagdown NH \end{smallmatrix} C_2H_5$ , of m.p. 251°–

252° C. (B) A mixture of, e.g., 1000 pts. of tetrahydronaphthalene and 15–20 pts. of aluminium chloride is heated for about 6 hrs. below 100° C. The product is washed with acidified water, and distilled. Benzene and unchanged tetrahydronaphthalene pass over in the first fractions, while octahydrophenanthrene, octahydroanthracene, and a polycyclic, hydroaromatic hydrocarbon distil respectively at 160°–170° C., 170°–180° C., and 230°–235° C. (11 mm.); these compounds on heating with sulphur or distillation with zinc dust yield respectively phenanthrene, anthracene, and an aromatic hydrocarbon of m.p. 168°–169° C. The octahydroanthracene has m.p. 72°–73° C., and is not identical with the compound previously described. The products or mixtures of the same are suitable for use as lubricants.—L. A. C.

See also pages (A) 339, *Still* (G.P. 333,216); *Lubricating oil* (G.P. 330,276); *Lubricants* (G.P. 330,970); *Asphalt substitute* (G.P. 330,650). 349, *Asphalt-like material* (G.P. 332,634).

#### IV.—COLOURING MATTERS AND DYES.

*Halogenated indigos; Halogenated nitrobenzaldehydes and* — L. C. Janse. Rec. Trav. Chim., 1921, 40, 285–317.

*p*-AMINO BENZALDEHYDE when brominated in solution either in dilute hydrochloric acid or in acetic acid gives an almost quantitative yield of 3,5-dibromo-4-aminobenzaldehyde. In this latter compound the amino group may be replaced more or less readily by chlorine, bromine, iodine, or hydrogen, and the products on nitration give substituted *o*-nitrobenzaldehydes, which may be converted by treatment with acetone and alkali into halogenated indigos (*cf.* Baeyer and Drewsen, Ber., 1882, 15, 775; 1883, 16, 2188). A whole series of such compounds is described. (*cf.* J.C.S., June.)—W. G.

*Auxochromes; Combined* — H. Kauffmann. Ber., 1921, 54, 795–802.

SUCH auxochrome-like groups of atoms as exhibit increased auxochromic character on account of the presence in them of true auxochromes are termed combined auxochromes. Their properties are examined in the particular instance of the dimethoxystyryl group, the *p*-nitrophenyl residue being used as chromogen. The utility of styryl in this connexion depends on the immediate attachment of its ethylenic linkage to the benzene nucleus, since substances such as 4-nitro-2,4'-dimethoxychalcone are only feebly coloured. The whole dimethoxystyryl complex is to be regarded as a new auxochromic unit. The combined auxochromes, like the simpler ones, are able to facilitate fluorescence. (*cf.* J.C.S., June.)—H. W.

#### PATENTS.

*Hydroxyazo dyes; Manufacture of* — G. T. Morgan, and British Dyestuffs Corp., Ltd. E.P. 160,848, 17.7.19.

DIAZOTISEN *o*-aminophenols, *o*-aminonaphthols, and their nitro, sulphonic, and carboxyl derivatives are coupled with 4,6-diamino-1,3-xylene, yielding products which dye oninal fibres brown shades in conjunction with metallic mordants.—L. A. C.

*Azo dye*. T. H. Leaming, Assr. to National Aniline and Chemical Co., Inc. U.S.P. 1,372,439, 22.3.21. Appl., 27.2.20.

A DISAZO dye is claimed, having the probable formula  $NH_2.C_6H_4.(4N_2)(2).C_6H_4.(1NH_2).(4)SO_3Na.(8)OH$ .

(7)N<sub>2</sub>C<sub>6</sub>H<sub>4</sub>(4)NHC<sub>6</sub>H<sub>4</sub>O, which yields *p*-phenylenediamine and triamino-8-naphthol-4-sulphonic acid on saponification and reduction with stannous chloride and hydrochloric acid, and dyes cotton, wool, natural and artificial silk greenish-black shades which can be developed on the fibre.

—L. A. C.

Condensation products resembling indophenol; Preparation of —, Act.-Ges. für Anilin-Fabr. G.P. 333,897, 28.11.16.

*p*-NITROSOPHENOL, *p*-nitroso-*o*-cresol, or *p*-nitroso-*o*-chlorophenol is condensed with a phenol alkyl ether which is not substituted in the para position to the alkoxy group, such as anisol, *o*-chloroanisol, phenetol, *o*-cresol ethyl ether, or resorcinol dimethyl ether, in the presence of sulphuric acid of about 60° B. (sp. gr. 1.71), or concentrated hydrochloric acid. The products separate from the acid solution as brown or red compounds, which are coloured blue by the addition of alkalis, dissolve in concentrated sulphuric acid to give blue solutions, and in aqueous sodium sulphide to give feebly coloured solutions which turn blue in the presence of air, and yield the corresponding leuco compounds on the addition of acids.—L. A. C.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cotton and other textile fibres; Action of sea-water on —. C. Dorée. Biochem. J., 1920, 14, 709—714.

FABRICS of cotton and silk are destroyed by immersion in sea-water for three weeks, wool lasting somewhat longer. This destructive action is, in the case of cellulose, due to micro-organisms, and not to oxygen, light, or the salts present. In its nature it resembles the "mechanical" breakdown of cotton sometimes observed under the "beetling" process (cf. Croas, J., 1920, 58 A). If cotton is acetylated to the monoacetate stage so that its structural qualities are preserved, the resulting material is very resistant to sea-water. Cellulose acetate silk withstands the action of sea-water for months.

—J. C. D.

Fibrous materials; Molecular structure of —. R. O. Herzog and W. Jancke. Umschau, 1921, 25, 53—54. Chem. Zentr., 1921, 92, II., 573.

FROM experiments with Röntgen rays it is concluded that in plant fibres the cellulose is present in a crystalline form disposed symmetrically with respect to the axis of the fibre. Artificial silk consists of an irregular felted mass of crystalline fragments of unchanged cellulose; artificial silk made from cellulose acetate is amorphous and consists of a mixture in which the cellulose acetate has undergone considerable degradation. Natural silk shows a crystalline character and a similar symmetrical structure to that of cellulose. Animal hair is amorphous, and probably consists of two or more substances. Starch and pure fats are crystalline.

—J. H. L.

Polysaccharides. III. Cellulose. P. Karrer and F. Widmer. Helv. Chim. Acta, 1921, 4, 174—184.

COMPARISON of the yields of cellobiose octo-acetate obtained by treating cellulose, cellobiose, methylcelloside, cellulose triacetate, cellobiose acetate, and methylcelloside acetate with acetic anhydride and sulphuric acid under similar conditions leads to the conclusion that cellulose must contain at least about 50%, and possibly more, of ready-formed cellobiose complex. Hess and Wittelschach's formula for cellulose (J., 1920, 512 A) cannot, therefore, be accurate, as it indicates only 33% of pre-formed cellobiose. Methylation of cellobiose by means of

alkali and methyl sulphate gives unsatisfactory results, but methylation of methylcelloside in this way gives an almost quantitative yield of hexamethylmethyloctoside. The latter may be further methylated, by means of dry methyl iodide and silver oxide, to heptamethyl-β(P)-methylcelloside, and hydrolysis of this by boiling 5% hydrochloric acid solution yields products from which β-tetramethylglucose may be isolated. (Cf. J.C.S., May.)

—T. H. P.

Cellulose. III. Asymmetric structure of cellulose and the influence of ammoniacal copper hydroxide [Schweizer's reagent] on the rotation of carbohydrates. K. Hess and E. Messmer. Ber., 1921, 54, 834—841.

LEVALLOIS' observation that solutions of cellulose in ammoniacal copper hydroxide solution are optically active is confirmed, and the activity is proved to be due to the cellulose content. Under slightly varying conditions the values  $[\alpha]_D^{25}$  = ca. -950° and -1000° are found, but accurate work is particularly difficult with the blue light. On the other hand, the "regenerated" cellulose is optically inactive in concentrated hydrochloric acid solution, as are also ethereal solutions of ethylcellulose, aqueous alkaline solutions of xanthate (viscose) cellulose, and jellies of cellulose in neutral salt solutions. Cellulose presents, therefore, a marked example of "latent asymmetry," thus resembling mannitol, and it appears that ammoniacal copper hydroxide behaves towards it in the same manner as boric acid or borax to the simpler carbohydrates. (Cf. J.C.S., June.)—H. W.

Cellulose. K. Freudenberg. Ber., 1921, 54, 767—772.

CELLOBIOSE octa-acetate can be isolated in yield corresponding with the presence of 35—36% of cellobiose in the original material in a single operation when cellulose is treated with a mixture of acetic anhydride and concentrated sulphuric acid at a low temperature, the operation requiring about 14 days. An indirect estimation of the loss of octo-acetate which occurs during the change makes it probable that cellulose is composed of cellobiose to the extent of more than 60%. This result is not in harmony with Hess's conception of cellulose (J., 1920, 512 A) as composed of pentaglycosidylglucose residues, but is in agreement with the hypothesis that it is built up of cellobiose residues arranged in a uniform polysaccharide chain of ten or more members. (Cf. J.C.S., June.)—H. W.

Wood cellulose [; Action of sodium hydroxide on —]. W. G. Harding. J. Phys. Chem., 1921, 25, 201—203.

THE product remaining after heating wrapping paper with 1% sodium hydroxide solution at 100° C. amounted to about 60% of the original material, although absolute equilibrium was not reached even after 30 hrs. treatment. With 6% sodium hydroxide the same result was obtained in about 6 hrs. Lignocellulose could be detected in the material after treatment.—W. J. W.

Pinewood; Content of soluble carbohydrates in —. S. Schmidt-Nielsen. Tekn. Ukeblad, 1920, 67, 354—355. Chem.-Zeit., 1921, 45, Rep., 32.

PINEWOOD felled in the spring was finely rasped, and after extraction with ether was repeatedly extracted by alternate boiling with water or 0.3% acetic acid and treatment with alcohol. By more than 20 successive extractions a yield of soluble matter was obtained equal to 12.04% of the original wood or 13.9% of the dry substance. In determinations of reducing sugar by Kjeldahl's method 1 g. of the dried extract, before inversion, reduced 0.1898 g. of copper, and after inversion with 2%

hydrochloric acid it reduced 0.6165 g. of copper. A tantially similar extract was obtained from wood felled in the winter, but the yield in this was only one-seventh of that mentioned above. Tossan determinations on the extract indicated at 23% of xylose.—J. H. L.

*Lignified cell membranes; Contribution to the knowledge of —.* P. Casparis. Pharm. Monats., 1920, I, 121—129, 137—146, 153—160. Chem. Zentr., 1921, 92, II., 584.

LIGNIFIED cell walls may be distinguished from those consisting of unaltered cellulose by means of a 15—40% solution of cobalt thiocyanate which colours lignified membranes blue. The reaction is probably due entirely to adsorption; it is more sensitive and trustworthy than Maule's permanganate reaction or the phloroglucinol-hydrochloric acid test.—J. H. L.

*[Sulphite] pulp; Automatic cooking control for chemical —.* C. H. Allen. Techn. Assoc. Pulp and Paper Ind., 1920, III, 19—21.

It is suggested that by controlling automatically the digestion of sulphite pulp, an increase in production will be obtained, together with an improvement in the quality. The control system described consists essentially of a power-driven steam valve, electrically connected with a steam flow meter on the digester steam line, an automatic relief valve, and relief strainers.—J. C. K.

*Sulphite and sulphate [pulp] fibres; Tests for unbleached —.* R. E. Lofton and M. F. Merritt. Techn. Assoc. Pulp and Paper Ind., 1920, III, 1—7.

For distinguishing between unbleached sulphite and sulphate (kraft) pulps, the use of a mixture of 1 pt. of a 2% aqueous solution of Malachite Green and 2 pts. of a 1% solution of Fuchsine (Magenta), is recommended. The solutions, which should be used not more than a few hours after being mixed, should be tested with a standard mixture of sulphite and sulphate fibres. The colour contrast not only enables the presence of one or both of these fibres to be detected, but is sharp enough to allow of an approximately correct estimate of the percentages of the two kinds of fibres to be made.—J. C. K.

*Paper; Measurement of translucency and opacity of —.* R. Fournier. Paper, Feb. 9, 1921, 32, 44.

THE translucency and opacity of paper can be measured by an apparatus in which the eye of the observer is replaced by a selenium plate which forms part of an electric circuit; the electrical resistance of the selenium varies according to the amount of light which it receives at the time the determination is made. By a slightly modified form of the apparatus the reflecting power of a sheet of paper may also be determined.—J. C. K.

*Strawboard factory waste waters; Gas from —.* A. Hermann. Het Gas, 1920, 40, 131—135. Chem.-Zeit., 1921, 45, Rep., 32.

A COMMISSION appointed by the Dutch Government recommends storage in settling tanks followed by irrigation treatment or filtration as the best and cheapest means of purifying the effluent from strawboard factories. In a pulp factory employing this process it has been found that in absence of air the effluent undergoes a fermentation due to rodlet bacteria which, at an optimum temperature of 35° C., produce a gas containing 23—30% of carbon dioxide and 70—77% of methane. One volume of effluent evolves 2 volumes of gas, which is collected in bell covers, conducted to a gas holder and employed for the production of electric current for power and lighting in the factory, and the surplus

is taken by the local gas works and mixed with coal gas in the proportion of 25%. An analysis of the gas showed carbon dioxide 24.7%, methane 64.3%, other hydrocarbons 0.6%, hydrogen 4.9%, oxygen 0.4%, nitrogen 5.1%; it contained traces of carbon bisulphide but no hydrogen sulphide or carbon monoxide.—J. H. L.

*Sulphite spirit.* Sieber. See XVIII.

#### PATENTS.

*Fibres suitable for spinning; Method for producing — from papyrus.* J. J. Gähler. E. P. 144,249, 31.1.20. Conv., 3.6.19.

PAPYRUS, which has been retted in the usual manner, is subjected to a mechanical alternating right and left-hand twisting movement whereby the fibres are loosened. The attached substances are then removed by brushing and combing, and also by means of a second retting treatment, if a very clean fibre is desired.—A. J. H.

*Wool; Process for producing spun material resembling — from viscose solutions.* Glanzfäden A.-G. E.P. 152,351, 13.11.19. Conv., 15.8.18. Addn. to 135,205 (J., 1921, 254 A).

In the preparation of viscose solutions, as described in the chief patent, aerial oxidation of the cellulose takes place. This is prevented by the addition of about 5% (on weight of cellulose) of sugar (preferably cane sugar) to the aqueous caustic soda solution in which the cellulose xanthate is dissolved.—A. J. H.

*Lace, fabrics and the like; Fireproofing, metallising, and waterproofing —.* A. Norweb. E. P. 160,627, 30.1.20.

LACE or fabric is padded with an emulsion formed from a solution of sodium silicate and sodium bicarbonate, a metallic powder, and a solution of rubber in naphtha and amyl acetate, and is then dried.—A. J. H.

*Artificial silk thread; Production of —.* G. F. J. Bouffe. E. P. 160,859, 3.9.19.

CELLULOSE threads which issue from a squirting nozzle are immediately drawn through a chamber which is exhausted of air or contains such gases as sulphur dioxide, carbon dioxide, etc., or through which a current of air is drawn, and are then wound on spools. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 28,733 of 1904, 14,087 of 1906, 11,729 and 24,707 of 1910, and 10,857 of 1915; J., 1905, 1299; 1906, 844; 1910, 1004; 1911, 126; 1916, 533.)—A. J. H.

*Cellulose fibres; Production of easily bleached —.* B. Possanner von Ehrenthal. G.P. 331,802, 8.8.19.

MATERIAL such as flax or hemp straw, jute, reeds, nettles, potato haulms, willow bark, or the like, with or without a preliminary mechanical treatment, is treated at 40°—50° C. with a 0.5 to 2% solution of an organic or inorganic acid or acid salt to hydrolyse the binding material, and is subsequently heated, under normal or increased pressure, with a weak alkaline solution containing a small proportion of an organic solvent such as alcohol, carbon bisulphide, acetone, petroleum, higher hydrocarbons, or the like. The individual fibres are separated from the fibrous product by steeping in a warm solution or emulsion of fatty or oleic acids, or their salts, esters, sulponic acids, or amides.—L. A. C.

*Bast; Production of — from fibrous plants.* Nessel-Anbau-Ges.m.b.H. G.P. 331,896, 2.2.18.

THE bast is loosened without destruction of its

natural ribbon or tubular form by the action on the plant (ramie, nettles, reeds, hops, flax, etc.) of water or a solution of sugar (molasses), and is subsequently separated into fibres by mechanical and chemical means.—L. A. C.

*Fibres from the needles of pines and other conifers; Production of textile* — R. Guttmann and J. Siegert. G.P. 332,096, 8.5.19.

THE dried needles are heated to 60°–70° C. in dilute mineral acid to loosen the epidermal silicious layer, and resinous material is removed from the fibres by subsequent treatment with a 3% solution of alkali. The alkaline resin solution can be employed for sizing paper.—L. A. C.

*Bast fibre plants; Retting* — P. Kraiss and K. Biltz. G.P. 332,097, 24.8.19.

THE addition of a small proportion of bicarbonates as well as chalk or other insoluble carbonate to the liquor accelerates the retting process, improves the quality of the fibre, and diminishes the formation of products with a repulsive odour.—L. A. C.

*Flax; Process for retting* — P. Kraiss. G.P. 332,514, 26.3.20.

THE raw flax is steeped in water or an alkaline solution and is stored moist in the dark at a moderate temperature to promote the formation of fungi, such as species of *Aspergillus*, and retting bacteria, such as *Plectridium pectinovorum*. After 3 to 4 days the retting is complete, and the material is dried at 80° C. or above to kill the fungi and bacteria.—L. A. C.

*Peat wool; Production of chemically pure, curly* — from peaty cotton-grass. O. Linker. G.P. 332,169, 26.10.18.

THE raw fibrous material, after preliminary treatment for  $\frac{1}{2}$  hr. at 50° C. in a 0.5% solution of an alkali, or sulphuric, hydrochloric, or hydrofluoric acid, or other suitable solvent, is rinsed and allowed to ferment for at least 4 hrs. in wooden vessels containing 1 kg. of malt or diastase per 1 cub. m. of water, or 0.5% acid solution, at 50°–60° C., or 11 kg. of yeast per 1 cub. m. of water at 30°–40° C. The fibres are then boiled for at least  $\frac{1}{2}$  hr. in an alkaline or acid solution, and are subsequently rinsed with cold water and bleached.—L. A. C.

*Fibrous plants; Process for the simultaneous production of textile fibres and paper pulp from* — Nessel-Anhau-Ges.m.b.H. G.P. 332,170, 16.8.19. Addn. to 328,596 (J., 1921, 211 A).

THE hydrocarbons or halogen derivatives of hydrocarbons employed in the process as described in the chief patent are of the same sp. gr. as the aqueous solution, and thus readily remain in suspension in the same. A sodium hydroxide solution of 5°–8° B. (sp. gr. 1.037–1.060) is recommended, in which case the material is treated for  $4\frac{1}{2}$  to 5 hrs. under 8 atm. pressure.—L. A. C.

*Waterproofing and sizing composition; Adhesive* — A. E. Craver. U.S.P. 1,373,412, 5.4.21. Appl., 8.6.20.

THE composition contains a protein, a suitable aqueous solvent, and a substance capable of liberating formaldehyde.—A. J. H.

*Impregnating agents [for fabrics, wood, leather, etc.]*. Farbenfahr. vorm. F. Bayer und Co. G.P. 302,531, 9.2.17.

HALOGEN derivatives of benzene homologues or of coal-tar hydrocarbons containing side-chains, are condensed with tar oils or their constituents (anthracene, naphthalene, benzene, etc.) or derivatives thereof. The oily or resinous products are very suitable for impregnating fabrics, paper yarn, wood, leather, etc., as they adhere firmly to the

material, are scarcely soluble in water, and possess preservative properties.—J. H. L.

*Impregnating agents; Removal of* — from fabrics. G. Bonwitt. G.P. 331,285, 18.7.18.

THE fabrics are extracted, in a suitable apparatus, with solvents of high b.p., such as ethyl lactate, acetylene tetrachloride, or furfural, or with mixtures of such solvents with others of low b.p., e.g., ethyl lactate and formate, acetylene tetrachloride and acetone, cyclohexanone and ethyl acetate, or furfural and ether, or with solvents containing substances which lower the viscosity of the material undergoing extraction; alcohol, for example, lowers the viscosity of solutions of cellulose esters.

—J. H. L.

*Wood or other substances containing cellulose; Treatment of* — for the purpose of obtaining cellulose and artificial resin, asphalt, lac, and the like. C. Claessen. E.P. 160,482, 17.10.19.

CELLULOSIC materials, such as wood, straw, grass, etc., are heated for a few hours at about 100° C. and under reduced pressure with phenol or other phenolic substances either in concentrated form or diluted with water, alcohols, benzol, or aliphatic hydrocarbons; 0.01% of hydrochloric acid (on weight of phenol) is added as a catalyst. The snow-white cellulose pulp produced is removed, part of the phenol is recovered by distillation, and lac or resin is obtained from the liquid residues by precipitation or distillation.—A. J. H.

*Cellulose compounds; Manufacture of* — Deutsche Celluloid Fabr. G.P. 332,203, 10.1.18.

MERCERISED cellulose is treated with chloroacetic acid, or its homologues, in the presence of alkali hydroxides. The alkali salts produced swell in hot or cold water, and finally dissolve to form clear, viscous solutions which on evaporation yield films of a material resembling glue or gelatin and inflammable with difficulty. The free cellulose-acetic acid is a white fibrous substance, insoluble in water, but soluble in alkaline solutions; with lead, copper, or aluminium oxides in the presence of water it forms insoluble salts. The alkali salts of cellulose-acetic acid and cellulose-propionic acid may be used as substitutes for gelatin.—L. A. C.

*Paper, posteboard, and the like; Process for sizing* — W. Merckens. E.P. 147,006, 6.7.20. Conv.; 7.12.15.

ANIMAL or vegetable size (10% on the weight of the fibre) may be directly worked up with half-stuff in the hollander if it be added in a gelatinous form and melts in the subsequent drying process. If necessary, formalin, chrome alum, or other hardening agent is added to the size.—A. J. H.

*Paper-making machinery [; Means for washing the woollen wet felts of]*. C. Walmsley and Co., Ltd., and J. Law. E.P. 160,861, 13.9.19.

*Drying coated or enamelled paper, waterproofed cloth, and such like; Apparatus for* — C. F. Howden. E.P. 160,940, 31.12.19.

*Fruit and vegetable refuse*. G.P. 331,005. See XIX.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

[Printing.] Oxidation by steaming a mixture of a chlorate and an aldehyde-bisulphite compound. A. Pellizza. Sealed Note 1469, 6.5.04. Bull. Soc. Ind. Mulhouse, 1920, 86, 640–641. Report by A. Lipp. *Ibid.*, 641.

THE reduction of a chlorate by an alkali bisulphite occurs in the cold with too great violence to be applied in printing, but when acetone-bisulphite or

formaldehyde-bisulphite is used, the chlorate is only reduced at about 100° C. and the reaction may be regulated by the presence of small quantities of certain salts. For example, by printing a mixture of a chlorate, acetone-bisulphite, and the salt of an aromatic amine, followed by steaming for several minutes, Turkey Red, chrome mordants, etc. are discharged and Aniline Black is developed. The fibre is not attacked more than is the case when the ordinary mixture of ferrocyanide and chlorate is used. Lipp confirms these claims and agrees that the mixture can be used as an oxidising agent for the development of Aniline Black and as an oxidising discharge. In the case of white discharges, the white is not as clear as that obtained by the use of chlorate, ferrocyanide, and tartaric acid.—F. M. R.

**Discharges; Coloured** — on azo-grounds with sulphozylate-formaldehyde and basic dyes. Justin-Mueller. *Sealed Note* 1931, 15.12.09. *Bull. Soc. Ind. Mulhouse*, 1920, 86, 636—637. Report by C. Sunder. *Ibid.*, 637—639.

The preparation of the fabric with potassium ferrocyanide (J., 1908, 320) complicates the process, as drying is necessary. In order to simplify the process and render it analogous to the usual antimony tannate method, it is modified by the omission of the preliminary preparation, and after steaming the fabric is passed through a potassium ferrocyanide bath. When New Methylene Blue N is used, fuller and brighter discharges are obtained with the modified zinc-ferrocyanide process than is the case with antimony tannate. Sunder reports that the process is worthy of attention in view of the brightness of the shade and the possibility it affords of producing rose-coloured discharges with Rhodamine.—F. M. R.

#### PATENTS.

**Dyeing and waterproofing [fabrics]; Process for** —. A. O. Tate. U.S.P. 1,374,122, 5.4.21. Appl., 20.4.20.

FABRIC is padded with the usual Aniline-Black liquor, aged, impregnated with sodium palmitate and an aluminium salt, and then subjected to an electric current while passing between electrodes, one of which is of aluminium.—A. J. H.

**Dyeing fur, hair and the like.** Chem. Fabr. Griesheim-Elektron. G.P. 334,012, 15.12.16.

Soft leathers and hair may be dyed shades fast to light by means of the mineral acid salts of amino- and imino-compounds and oxidising agents. The dissociation of the mineral acid is diminished by the addition of neutral salts. By the addition of copper sulphate, a bichromate, sodium perchlorate, and hydrochloric acid to aniline hydrochloride dissolved in a 10% sodium chloride solution, a grey shade fast to light is obtained. Under similar conditions, monobenzylaniline hydrochloride gives a bluish-grey; monobenzyl-2.6.-toluenediamine hydrochloride gives a bright yellowish-grey, and mono- $\beta$ -naphthyl-2.6.-toluenediamine hydrochloride gives a bright fawn shade.—A. J. H.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

**Nitrous acid; Decomposition of** —. [Detection of nitric acid in presence of nitrous acid.] E. Oliveri-Mandalà. *Gazz. Chim. Ital.*, 1921, 51, I., 138—140.

HYDRAZOIC acid and nitrous acid react quantitatively according to the equation,  $\text{HNO}_2 + \text{HN}_3 = \text{H}_2\text{O} + \text{N}_2\text{O} + \text{N}_2$ , no trace of nitric acid being formed. This reaction serves for the detection of nitric acid in presence of nitrous acid, other methods for such

detection yielding uncertain results owing to the formation of appreciable traces of nitric acid by partial decomposition of the nitrous acid. The test is carried out as follows:—Either a few c.c. of dilute hydrazoic acid solution or a little of its sodium salt is added to the solution containing nitrous and nitric acids, this being first acidified with acetic acid when the nitrous acid is present as salt. The liquid is boiled to expel, so far as possible, the excess of hydrazoic acid, a portion of the liquid being then tested with an acetic acid solution of naphthylamine and sulphanic acid to ascertain if all the nitrous acid has been destroyed, and the remainder examined for nitric acid.—T. H. P.

**Reduction by metals in acid solutions. I. Reduction of acid ferric sulphate solutions by zinc and magnesium.** S. Sugden. *Chem. Soc. Trans.*, 1921, 119, 233—238.

QUANTITATIVE determination of the "percentage reduction" (that is, the percentage of that theoretically possible if no hydrogen were evolved) of acid ferric sulphate solutions, containing varying concentrations of ferric oxide and of sulphuric acid, by means of zinc and magnesium establishes the fact that with both metals increase in the concentration of ferric salt is accompanied by an increase in the percentage reduction, and also the further addition of sulphuric acid to a nearly neutral solution causes a decrease in the percentage reduction. Whilst, however, with magnesium this latter decrease continues progressively as the acid concentration increases, with zinc a minimum is reached, after which the percentage reduction increases rapidly and a new type of reaction apparently sets in, the zinc becoming dull grey in colour, and dissolving much more slowly with a scarcely perceptible evolution of gas. This peculiar behaviour of zinc is most marked with the solutions containing the higher concentrations of ferric oxide.—G. F. M.

**Fluorine; Preparation of** — from molten potassium bifluoride. F. Meyer and W. Sandow. *Ber.*, 1921, 54, 759—766.

THE method is a development of that proposed by Argo and others (J., 1919, 413A). A cylinder of Acheson graphite, which acts as crucible and cathode, is closely surrounded by an electrically-heated copper cylinder. The anode, also of Acheson graphite, is made wider at the lower than at the upper end, and is provided with six vertical grooves to increase the amount of surface exposed. The diaphragm consists of a copper cylinder to the lower end of which four conical copper rings are successively attached and, finally, a reversed ring fastened to a horizontal piece of copper foil. Potassium bifluoride, which gives a thin, clear melt at 240° C., is used as electrolyte, this having proved superior to mixtures of the bifluoride with sodium fluoride or hydrogen fluoride, lead or strontium fluorides in point of fluidity and loss of hydrogen fluoride. The crucible is slowly heated to a temperature not exceeding 250° C. and, since the molten salt obstinately retains traces of water, the electrolysis is started with a small current which is continued till the water is completely decomposed, after which the electrolysis proper is commenced. The evolved fluorine is contaminated with hydrogen fluoride, which can be removed by passage over granular sodium fluoride, and, initially, with ozone, which disappears after  $\frac{1}{2}$ — $\frac{3}{4}$  hr. The electrodes are but little attacked, and the gas does not contain more than 0.2% of carbon tetrafluoride. The current yield is more than 75% of that theoretically possible. (Cf. J.C.S., June.)—H. W.

**Phosphorus; Oxidation and luminescence of** —. H. B. Weiser and A. Garrison. *J. Phys. Chem.*, 1921, 25, 61—81.

THE reaction between phosphorus and oxygen pro-

ably takes place in the vapour phase, and hence the velocity of reaction,  $V$ , may be expressed by the equation  $V = kC_p^x C_o^y$ . Since  $C_p$  will be constant in presence of solid phosphorus, the velocity should be proportional to some power of the oxygen concentration  $V = kC_o^y$ . This, however, is not the case below  $25^\circ \text{C}$ , and above a certain partial pressure of the oxygen, owing to the formation of a film of oxide on the phosphorus. If the oxygen pressure is great enough, the reaction takes place near the surface of the phosphorus, which will be coated with a film of oxide. By decreasing the partial pressure of the oxygen, this film is destroyed. The same effect may be obtained by increasing the rate of evaporation of the phosphorus. The pressure (boundary pressure) above which a protecting film prevents the oxidation and the vaporisation of the phosphorus is not constant. The intermittent luminescence in the narrow critical region between the boundary pressure and the glow pressure is probably due to the periodical break-down and reformation of this film.—J. C. K.

*Melting point of potassium chlorate.* Carpenter. See XXIII.

*Sodium sulphantimonate.* Langhans. See XXIII.

#### PATENTS.

[Lead] oxides; Method of converting metallic lead into —. J. A. Thibault. E.P. 137,288, 29.12.19. Conv., 30.12.18.

GRANULATED lead is heated to  $250^\circ \text{C}$  for 6 hrs. in a closed rotating drum with compressed air at 60 lb. per sq. in. pressure and a small quantity of water vapour, whereby it is converted into litharge of a yellow-ochre colour and of an extra fine texture. Red lead may be produced in the same apparatus by increasing the temperature to  $425^\circ \text{C}$ , as soon as all of the lead has been converted into litharge, and continuing the heating for a further period of 4 hrs. The product is a bright red impalpable powder containing over 30% of lead peroxide.—A. R. P.

*Electrolysing vat [for making chlorine].* H. Loeb. U.S.P. 1,372,176, 22.3.21. Appl., 10.1.19.

AN electrolysising vat for making chlorine consists of a vessel of glazed pottery surrounded by a wooden casing, the intervening space being packed with asphalt.—J. S. G. T.

*Calcium carbonate; Manufacture of —.* L. Roon. Assr. to The Equitable Trust Co. U.S.P. 1,372,193, 22.3.21. Appl., 26.4.19.

MILK of lime is atomised, and the resulting mist allowed to react with carbon dioxide.—C. I.

*Calcium carbonate; Manufacture of —.* J. Faldner. G.P. 334,014, 26.11.19.

QUICKLIME is dissolved in a solution of an ammonium salt and impurities are removed by precipitation and filtration. The liquid is then saturated with carbon dioxide under pressure to precipitate calcium carbonate and regenerate the original ammonium salt for use again in the process.

—A. R. P.

*Aluminium chloride; Production of —.* E. C. Baum and De W. O. Jones, Assrs. to Armour Fertilizer Works. U.S.P. 1,372,332, 22.3.21. Appl., 24.3.20.

ALUMINIUM nitride is subjected to the action of chlorine.—C. I.

*Arsenic trioxide; Process of purifying —.* C. P. Linville, Assr. to American Smelting and Refining Co. U.S.P. 1,372,443, 22.3.21. Appl., 21.7.16. Renewed 18.12.19.

COLOURING matter due to selenium is removed by

mixing with a reagent capable of forming a non-volatile selenide at a comparatively low temperature and separating the arsenious oxide by sublimation.—C. I.

*Zinc oxide; Manufacture of —.* W. L. Coursen, Assr. to The New Jersey Zinc Co. U.S.P. 1,372,486, 22.3.21. Appl., 28.6.20.

IN the production of zinc oxide by the retort volatilisation process, a substantial quantity of molten zinc is maintained in the retort during the whole of the operation, volatilisation losses being made good by the addition of solid zinc to the contents of the retort from time to time.—A. R. P.

[Zinc] oxide; Manufacture of French —. J. A. Singmaster, Assr. to The New Jersey Zinc Co. U.S.P. 1,372,462, 22.3.21. Appl., 31.3.20.

THE contents of the retorts in which the zinc is volatilised are kept nearly constant by the addition from time to time of molten zinc.—A. R. P.

*Bichloride of mercury; Manufacture of —.* C. Schantz. U.S.P. 1,373,357, 29.3.21. Appl., 6.12.20.

MERCURY is passed through an atmosphere of chlorine into a bath of a liquid which does not readily absorb chlorine.—A. R. P.

*Fixation of gases [atmospheric nitrogen]; Process for the electric —.* G. T. Southgate. U.S.P. 1,373,639, 5.4.21. Appl., 22.11.20.

STREAMS of gas or gases are passed through porous electrodes between which an electric arc is established.—J. S. G. T.

*Calcium cyanamide; Production of — from carbide and nitrogen.* A.-G. für Stickstoffdünger. G.P. 299,142, 15.10.15.

CALCIUM carbide and calcium chloride or fluoride are ground together as finely as possible and the mixture, in layers 40–100 cm. deep, is exposed to nitrogen at a lower temperature than has been necessary hitherto, e.g.,  $650^\circ$ – $700^\circ \text{C}$ , when calcium chloride is used and  $700^\circ$ – $750^\circ \text{C}$  when fluoride is used.—J. H. L.

*Calcium cyanamide; Process and furnace for the production of high-grade crude —.* Bayerische Stickstoff-Werke A.-G. G.P. 326,613, 15.3.18.

A NUMBER of small charges are arranged together in a furnace and brought to the ignition point simultaneously. The nitrogen gas necessary for the reaction is brought into the furnace through channels in the wall. Other spaces in the wall are filled with powdered or granular insulating material.—J. H. J.

*Gases; Treatment of — for removal of hydrogen sulphide.* Badische Anilin- u. Soda-Fabrik. G.P. (a) 299,163, 2.7.16, and (a) 331,322, 22.10.16.

(A) GASES are freed from hydrogen sulphide by treating them with a solution of a compound of iron and oxygen in presence of an alkali, the reduced iron compound being subsequently regenerated by means of air or oxygen. In the case of gases containing little or no carbon dioxide, admission of carbon dioxide, which may be introduced in the form of an alkali carbonate, assists the re-oxidation of the iron compound. A suitable gas-washing solution is prepared by treating 300 kg. of potassium carbonate in 600 l. water with 90 kg. of oxalic acid and 135 kg. of 40% ferric chloride solution, and diluting the product to 1 cub. m. (s) The washing solution may contain oxalic acid together with tartaric acid, and a suitable mixture consists of 200 kg. of potassium carbonate, 20 kg. of crude acid potassium tartrate, 10 kg. of oxalic acid, and 90 kg. of 40% ferric chloride solution, diluted to 1 cub. m.—W. J. W.



*Alkali cyanide solutions; Decomposition of* —. W. Salge und Co., Techn. Ges. m. b. H. G.P. 332,297, 9.4.20.

THE alkali cyanide is decomposed, and the hydrocyanic acid is removed by passing a current of carbon dioxide into the solution, which contains at least 50% of alkali carbonate. Hydrocyanic acid is distilled without decomposition when carbon dioxide is passed into an alkali cyanide solution containing 50–60% of alkali carbonate at 90° C.

—C. A. C.

*Calcium hydride; Process for making* —. K. Kaiser. G.P. 333,305, 11.2.20.

HYDROGEN is passed over calcium chloride at 600°–700° C. until no further evolution of hydrogen chloride takes place. The residue of pure calcium hydride may be utilised for the hydrogenation of liquid fats and crude petroleum, or for the conversion of acetaldehyde into alcohol as well as for the production of ammonia from the air.—A. R. P.

*Ammonia soda; Manufacture of* —. J. Delmar. G.P. 333,748, 19.11.16.

THE lime required for the regeneration of ammonia from the ammonium chloride liquors is transformed into calcium cyanamide before being used in the process, whereby further quantities of ammonia are developed as well as a certain amount of carbon dioxide, which is utilised in the first stage of the ammonia soda process. Any excess of ammonia over that required in the latter process may be converted into a suitable salt. 1000 kg. of calcium cyanamide yields 800 kg. of sodium carbonate.—A. R. P.

*Sulphur; Process and apparatus for burning* — in easily combustible substances, e.g., spent oxide. Chemische Industrie A.-G. G.P. 333,818, 25.8.15.

THE material is charged into a multiple-hearth furnace, underneath the top hearth, through a special charging apparatus, the lower opening of which is kept closed by building up the charge around it. This apparatus is provided with an arrangement to prevent the material sintering and sticking in the neck of the charging cone.—A. R. P.

*Potassium salts containing kieserite; Process for utilising the sulphur content of crude* —. J. Kiermayer, and Hannoversche Kaliwerke A.-G. G.P. 333,963, 12.11.19. Addn. to 310,072 (J., 1920, 406 A).

THE operation of heating the fused salts with wood charcoal is carried out while passing a current of steam through the mass. Instead of charcoal, liquid or solid hydrocarbons may be used in the process.

—A. R. P.

*Carbon; Production of pure* —. F. C. Dyche-Teague. E.P. 160,561, 23.12.19.

COKE is converted into carbon monoxide by interaction in a gas-fired retort with carbon dioxide produced in a subsequent stage of the process. The carbon monoxide is decomposed into carbon and carbon dioxide by the catalytic action of finely divided iron or ferric oxide in a separate air-cooled chamber after filtration through firebrick. The carbon deposit and catalyst are blown out of the chamber and separated by a magnetic field or by gravitation.—C. I.

*Hydrozides; Manufacture of metal* —. M. Buchner. E.P. 16,597, 24.11.15.

SEE U.S.P. 1,337,192 of 1920; J., 1920, 488 A.

*Oxidation of hydrogen sulphide*. G.P. 331,287. See IIA.

*Hydrating lime*. E.P. 160,556. See IX.

## VIII.—GLASS; CERAMICS.

*Glass; Heat absorption in* —. A. Q. Tool and C. G. Eichlin. J. Opt. Soc. Amer., 1920, 4, 340–363. Chem. Zentr., 1921, 92, II., 698–699.

ON heating glass of constant heat conductivity through a certain well-defined temperature interval some change takes place in the structure of the glass, accompanied by an absorption of heat. An apparatus is described for measuring this heat absorption by comparing the temperatures of a powdered sample of the glass and a sample of clay under the same conditions of heating. The temperature at which the absorption commences varies from 460° to 610° C., and that at which it attains its maximum is 30°–40° C. higher, according to the kind of glass tested. The maximum in the case of quartz is at 580° C. It is shown that on annealing glass not only must the tension be equalised in all parts, but the structure should be made homogeneous; further, annealed glass may undergo a slow transformation into the stable crystalline phase at ordinary temperatures, which gives rise to volume changes, and consequently renders such glass unsuitable for thermometers.

—A. R. P.

*Refractory materials; Behaviour of* — under load at high temperature. K. Endell. Stahl u. Eisen, 1921, 41, 6–9.

THE behaviour of small cylinders, 50 mm. long and 50 mm. diam., when heated to various temperatures under a pressure of 1–8 kg. per sq. cm. was investigated. The test-pieces were heated in an electric resistance furnace, the pressure—usually 1 kg. per sq. cm.—being applied by a weighted lever through specially hard electrode carbons. The temperature of the test-pieces was measured by a Holborn-Kurlbaum optical pyrometer. The test-pieces were heated so that the maximum temperature was attained in about 4½ hrs, but the results were not appreciably different when only half this time was required. According to their behaviour when heated under a pressure of 1 kg. per sq. cm., refractory materials may be divided into four groups:—(1) Fireclay bricks with a refractoriness of about cone 33 (1730° C.) began to soften at 1300° C., a 50 mm. cylinder being reduced to 30 mm. in a test lasting 4½ hrs., in which the maximum temperature reached was 1500° C. Bricks containing very plastic clay softened at 1150° C. The deformation was uniform, the sides of the cylinder bulging as its height was diminished. (2) Magnesite bricks with a refractoriness above 2000° C. began to soften at 1500° C. on account of the binding agent. A 50-mm. cylinder was reduced to 10 mm. in height in a test lasting 4½ hrs., the maximum temperature attained being 1650° C. The hot cylinders were feebly plastic and broke at the sides under compression. (3) Silica bricks with a refractoriness of cone 35 (1750° C.) did not soften below 1650° C., but broke in pieces in 4½ hrs. at a maximum temperature of 1680° C. The actual deformation was small. (4) Carbon bricks with a refractoriness above 2000° C. did not soften under pressure, and apart from a slight loss in volume (about 4%), due to some of the carbon burning away, the cylinders were not affected in a test of 5 hrs., in which a temperature of 1720° C. was reached.—A. B. S.

## PATENTS.

*Glass and vitreous materials; Manufacture of* —. A. A. Kelly and B. D. Jones. E.P. 160,495, 20.11.19.

IN mixtures for making glass and vitreous materials sodium pentaborate is substituted for borax or boric acid, the necessary adjustment of the alkali content being made in any suitable manner. A more homo-



geneous product is secured with an absence of striæ and air bubbles.—H. S. H.

[Glass] annealing furnace or leer. W. O. Amsler. E.P. 160,710, 12.7.20.

A FURNACE has its annealing chamber heated by the hot products of combustion circulated through a number of passages from combustion chambers placed over the top of the muffle. A heating zone is created in which the temperature is maintained above the critical temperature of the articles to be annealed, and a cooling zone which has a predetermined uniform temperature gradient. The articles pass through the muffle from the high temperature zone through the region of decreasing temperature.—H. S. H.

Opaque glazes, enamels, and glasses; Production of white ——. H. Sachse. G.P. 331,682, 20.7.18.

WHITE opaque glasses, enamels, and glazes are made by means of natural compounds of zirconium, tin, and beryllium in combination with fluorine compounds, such as alkali fluorides or fluosilicates, fluorspar, and either natural or artificial cryolite, in sufficient quantity to prevent the production of colour by the impurities in the other ingredients. The large proportion of fluorine compounds ensures good covering power, ready fusibility, high gloss, and great resistance to chemicals and heat.

—A. B. S.

Plastic masses; Manufacture of — and new industrial products obtained therefrom. L. Gauthier. E.P. 145,514, 22.6.20. Conv., 13.4.19. Addn. to 128,905 (J., 1920, 450 A).

A PLASTIC mass which is capable of being run whilst hot, for the manufacture of hollow bodies, which are unbreakable and capable of being washed (such as vases, dolls' heads, etc.), is prepared by incorporating about 60% of talc with about 30% of gelatin and about 10% of hide glue. The hide glue is melted in a water bath, and the gelatin caused to swell by soaking in water, before being used in the mixture, which is then melted and cooled to form a paste. Slices are cut from the paste, melted in a water bath, and the fluid mass run into a mould and allowed to cool until the desired thickness is attained before pouring out the excess paste. After drying, the hollow body is polished, washed in a solution of Castille soap, and finally treated in a solution of alum and 10% formol.

—H. S. H.

Plastic masses; Process for the production of — from matter non-plastic by nature, as refractory oxides etc. E. Podszus. G.P. 326,841, 13.7.13.

DURING the grinding process a colloidal solution is produced within the mass either by the known methods of colloid chemistry or by chemical action. For example, at least two non-plastic materials of different basic properties, as alumina and silicic acid, are brought into the most intimate contact with one another by grinding together for a long time in presence of a dispersion medium. The proportions of the two substances are so chosen that sufficient quantities of a colloidal solution are formed to enable the mass to be cast.—J. H. J.

Refractory material. E. D. Frohman. U.S.P. 1,372,016, 22.3.21. Appl., 8.12.19.

A REFRACTORY material comprises a mixture of finely-divided dry fireclay, silicious material, and a vegetable compound capable of forming a binder when mixed with water.—H. H.

Refractory brick. C. W. Berry, Assignor to Laclede-Christy Clay Products Co. U.S.P. 1,373,854, 5.4.21. Appl., 4.4.18.

THE process consists in mixing raw magnesite

and bauxite with water, calcining the mixture at a temperature sufficient to expel moisture and gas, re-grinding the product, mixing it with water, forming it into bricks and burning them to produce a hard refractory material.—A. B. S.

Tunnel-kiln. G. E. Norman. U.S.P. 1,372,773, 29.3.21. Appl., 17.2.20.

THE walls and crown of a tunnel-kiln of the customary shape are provided, throughout their full length, with longitudinal air-passages leading into an "exhaust air directing compartment."

—A. B. S.

Tunnel furnace with reversible regenerative heating. H. Koppers. G.P. 331,703, 14.9.18.

THE furnace is designed for heating refractory materials or iron and steel billets with maximum thermal efficiency, by exposure to the highest temperature just before leaving the furnace. The preheated gas and air are brought together at this point and burn while moving counter to the goods in the furnace. Two parallel tunnels are employed.

—H. J. H.

Ceramic materials; Grouped direct-fired furnaces for ——. H. Herda. G.P. 326,389, 23.4.19. Addn. to 324,977 (J., 1920, 821 A).

THE waste gas from a fired furnace and the hot air from a cooling furnace are conducted through flues beneath the furnaces, and are mixed and conducted into the firing duct of the furnace which is to be heated next and into the latter furnace itself. By this means any chamber not in use in the group may be fired as a separate furnace, whether for annealing, melting, or glazing, without interfering with the working of others.—J. H. L.

Ceramic mass of great strength; Production of an easily fusible porcellaneous ——. H. Becker, H. Fehring, and H. Johnke. G.P. 332,578, 21.9.18.

A READILY fusible porcellaneous ceramic mass of great strength is made by adding to an alkali-lime glass batch a quantity of fluorspar at least equal to the quantity of silica. The sand may be partly replaced by silicate rock, ashes, or slags. The high fluorspar content makes the fused mass very mobile and prevents the material from being sensitive to sudden changes in temperature, so that large articles do not need prolonged annealing. The ware is opaque.—A. B. S.

Heat treatment of articles [glass bulbs]; Method of — and apparatus therefor. Corning Glass Works, Assees. of J. Bailey. E.P. 140,374, 17.1.20. Conv., 17.3.19.

Glass; Manufacture [drawing] of —. F. J. Brougham. From Virginia Plate Glass Corp. E.P. 141,737, 15.4.20.

## IX.—BUILDING MATERIALS.

### PATENTS.

Refractory and insulating products; Manufacture of —. G. L. Dimitri and J. E. Delaunay. E.P. 142,513, 1.5.20. Conv., 16.7.18.

HARD, refractory, and insulating products are made by compressing a mixture of hard powdered materials (particularly magnesium silicate and multiple silicates) in a press box from which the air is exhausted. A steel plate or washer slightly smaller than the internal diameter of the press-box is placed on the powdered material, and the press-box is evacuated through an aperture in the airtight cover whilst the material is being compressed by a rising plunger. When the steel plate is

pressed against the aperture the compression is complete. The pressed pieces are subsequently burned, and are said to be exceptionally hard.

—A. B. S.

*Porous material [for building and insulation]; Manufacture of —.* Det Norske Aktieselskab for Elektrokem. Ind. Norsk Industri-Hypotekbank, E.P. 143,500, 6.5.20. Conv., 16.5.19.

A MATERIAL of uniform porosity, suitable for building and insulation purposes, is made by blowing air or steam into molten slag, stirring vigorously so as to convert the slag into a foam, cooling the foam rapidly by passing it between water-cooled rollers until it has a temperature at which its porosity remains constant and the mass remains plastic, compressing the mass into the desired shape in moulds, and then cooling it further by gas or air under pressure.—A. B. S.

*Oil-proof concrete bodies and method of making same.* J. Marcussen. E.P. 145,673, 30.6.20. Conv., 1.7.18.

HOLLOW bodies made from concrete, cement, mortar, etc., are coated with an artificial resin obtained by acting with formaldehyde on phenols or other cyclic compounds. After exposure to the air for a certain time the coating is rendered insoluble, and in this state permanently resists the action of mineral or fatty oils.—H. S. H.

*Time; Apparatus for the hydration of — and for similar purposes.* E. R. Sutcliffe. E.P. 160,556, 22.12.19.

THREE or more cylinders are arranged one above the other with their axes parallel. A shaft carrying vanes is mounted axially within each cylinder, and on rotation causes the material to be hydrated to move slowly along the cylinders. The material is fed into one end of the top cylinder and passes successively through the cylinders. Water is sprayed into the first cylinder, and the others are provided with steam jackets. The product is delivered completely hydrated and in a dried condition.—H. S. H.

*Roads; Manufacture of compositions for covering — and for like purposes.* M. Monnoyer and H. T. E. Kirkpatrick. E.P. 161,061, 17.3.20.

A COMPOSITION for covering roads similar to that mentioned in E.P. 4349 of 1914 (F.P. 466,878; J., 1914, 965) is made by incorporating 0.6–1% of fibrous material, such as sawdust, impregnated with tar and bitumen, with a concrete mixture containing 2000–2250 kg. of solid matter.—A. B. S.

*Cement; Manufacture of slow-setting —.* F. Ferrari, Assr. to Bombini Parodi-Delfino. U.S.P. 1,372,015, 22.3.21. Appl., 23.8.20.

A RAW mixture of the ordinary type used for Portland cement hut having a ratio of  $\text{Fe}_2\text{O}_3$  to  $\text{Al}_2\text{O}_3$  between 1 and 1.563 is converted into slag to prepare a slow-setting cement free from the binary calcium compounds of alumina or iron generally used.

—H. H.

*Wood-impregnating retorts; Condenser and separator for —.* J. H. Dunstan and R. A. Davis. U.S.P. 1,374,069, 5.4.21. Appl., 13.10.19.

IN order to create rapidly a high vacuum in a wood-impregnating retort the latter is connected with a barometric condenser. A pipe leads from the condenser into the side of a barometric separator, and another pipe connects the top of the separator with a vacuum pump. A discharge pipe leads downwards from the bottom of the separator and has its lower end submerged in liquid in a hot well, whereby the separator is able to discharge constantly whilst the vacuum is maintained.—A. B. S.

*Building material; Process for the production of — into which nails can be driven.* A. Hambloch. G.P. 326,963, 23.5.18.

A MIXTURE of coarse-grained volcanic sand, tufa, or pumice with a calcareous and fine-grained silicious cement, is shaken to obtain the highest possible density. The moulded articles are hardened with steam under pressure.—J. H. J.

*Lump plaster; Manufacture of —.* G. Polysius. G.P. 330,065, 12.9.19.

RAW gypsum is heated in the annular space between two inclined, concentric cylinders which are heated both externally and internally.—A. B. S.

*Shipbuilding material; Production of —.* S. Rosenstein. G.P. 331,674, 23.1.19.

A LIGHT material for shipbuilding etc. is made of a pulverised mixture of "1 pt. of cement, 10–25% of lime, and 20–35% of a white silicious and acid-resisting magnesium silicate of the talc group," which after being mixed with porous materials is allowed to stand and finally is mixed with sand or other aggregate and water. The product is so elastic that it can be used to produce richer mixtures than usual without risk of cracking, and it has an extraordinary toughness and a great resistance to blows.—A. B. S.

*Slag wool; Production of waterproof articles from —.* H. Brunn. G.P. 331,675, 9.9.19.

SLAG wool is converted into "flocks," the adventitious grains separated by a current of air, and the short fibres and sand grains removed by a sieve. The long fibres aggregate to balls, which are either mixed with a fusible waterproofing material and moulded hot or are placed in a perforated drum and coated by immersion in molten pitch, asphalt, or a resinous mixture, and afterwards shaped in moulds under light pressure.—A. B. S.

*Light porous bricks; Process for hardening —.* C. H. Schol. G.P. 332,294, 21.6.18.

LIGHT porous bricks are first exposed to air and are afterwards hardened by exposure to steam under high or low pressure. The preliminary aeration prevents ill effects otherwise produced on exposing such bricks to steam.—A. B. S.

*Mortar material; Production of — from dolomite in a rotary tube furnace.* F. M. Meyer. G.P. 332,583, 6.8.12.

THE dolomite is hurned for so long and at such a high temperature that the magnesium carbonate is wholly and the calcium carbonate about half converted into oxide.

*Lime-sand bricks; Production of — by means of raw chalk or quarry waste and clay.* H. Servange. G.P. 332,631, 25.6.14.

LIME-SAND bricks are hardened in brick kilns at 530° C. in a current of steam and carbon dioxide, the latter being advantageous in the hardening of monocalcium hydrosilicate. The fired bricks harden still further on exposure to air.—A. B. S.

*Artificial stone resembling asphalt; Production of —.* A. Flexer. G.P. 332,634, 5.11.18.

IN the manufacture of resin acids for soap making from wood tar by oxidation, a pitch is obtained as a by-product. Artificial stone, resembling asphalt, of great resistance and strength, is made by washing, purifying, and drying this pitch and then mixing it, in the molten condition, with 4–5 times its volume of silica, sand, or other aggregate, and allowing the mixture to cool in sheets or moulds.

—A. B. S.

*Cement mix; Preparation of — prior to burning.* G. Polysius. G.P. 332,705, 4.12.19.

Hot cement clinker (preferably from a rotary kiln) is mixed with raw cement mix or cement slurry, and later is separated therefrom by means of sieves. This enables the heat in the clinker to be used in preheating the raw material, and the usual cooling drums are rendered unnecessary.—A. B. S.

*Bricks from ashes and slaked lime; Production of —.* G. de Bruyn. G.P. 332,755, 14.6.19.

BRICKS are made of a mixture of lime, ashes, plastic clay, and water, which is moulded like concrete. The bricks "ring" when struck, are waterproof, and show little "scum." The strength of the bricks and the facility with which nails may be driven into them may be varied by the addition of porous materials such as sawdust, pumice, etc.

—A. B. S.

*Impregnating agents.* G.P. 302,531. See V.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Malleable iron; Influence of manganese on the mechanical properties of —.* E. Leuenberger. Stahl u. Eisen, 1921, 41, 285—287.

A HIGHER content than 0.4% Mn is permissible in malleable cast iron, the tensile strength of which increases with the manganese content. Up to about 1% manganese has no influence on the elongation; with higher percentages the elongation diminishes. The tensile strength decreases with the period of annealing while the elongation increases. The longer the time of annealing, the higher may the manganese content be without unfavourably affecting the elongation.—T. H. Bu.

*Fluorspar; Addition of — to Martin [open-hearth steel] furnaces.* S. Schleicher. Stahl u. Eisen, 1921, 41, 357—361.

If fluorspar is added to a Martin slag, it will only be decomposed up to a certain limit, lying between 2 and 2.5% of calcium fluoride. The fluorspar addition at first drives off silicic acid from the slag. This silicic acid is, however, continually renewed from the lining of the furnace. Fluorspar has also a desulphurising action, the sulphur being driven off and the renovated slag taking up more sulphur from the bath.—T. H. Bu.

*Electric steel arc furnaces; Regulation of — using movable electrodes.* W. G. Mylius. Trans. Amer. Electrochem. Soc., 1921, 213—225. [Advance copy.]

THE different types of automatic electrode regulators are described, including a newer type which operates with greater precision and higher speed than earlier forms. The electrode is moved at high speed until the current per phase is within 15%, on either side, of its normal value, at which point the continuously running stage is replaced by a slower vibrating motion.—C. A. K.

*Steel and iron; Gasometric determination of carbon in —.* H. Burkardt. Chem.-Zeit., 1921, 45, 342.

IN adapting Strohlein's apparatus (cf. J., 1916, 1085) to the gasometric determination of carbon in steel, the author prefers to use a furnace fitted with carborundum supports, to dispense with the condenser, and to use ordinary wash-bottles for the oxygen in place of those supplied with the apparatus.—W. F. S.

*Iron and steel; Influence of copper on the physical properties of —.* E. A. and L. T. Richardson. Chem. and Met. Eng., 1921, 24, 565—567. (Cf. J., 1920, 752 A.)

THE addition of even small amounts of copper to iron causes brittleness in forging, but this effect is produced only within a certain range of temperature, above or below which the alloy can be successfully forged. This interval during which "red-shortness" occurs increases with the percentage of copper in the alloy; thus iron containing 4.66% Cu can be forged only at a very low temperature. The phenomenon is probably due to an intergranular film of copper or of one of its alloys or compounds. By the addition of manganese or chromium brittleness is overcome, but the amount added must be proportional to the percentage of copper in the alloy; 0.32% of manganese added to an alloy containing 0.48% Cu removed brittleness, whilst the same amount added to an alloy with 0.92% Cu proved insufficient. The smaller liability of steel to brittleness is explained by its manganese content. The cold-working properties, such as filing, grinding, chiselling, and bending, of iron low in carbon, are not impaired by the presence of copper up to 5%. Manganese makes iron harder, and if the amount exceeds 2% the resulting alloy is too brittle and hard for working; the same proportion of chromium causes hardness and toughness but not brittleness.—W. J. W.

*Iron and steel; Anomalies encountered in a study of immersion tests of —.* A. S. Cushman and G. W. Coggeshall. Trans. Amer. Electrochem. Soc., 1921, 249—267. [Advance copy.]

A LARGE number of tests by the immersion of plates in different corroding solutions tend to show that copper-bearing iron is less resistant to corrosion than iron free from copper. After immersion in dilute (5%) mercuric chloride solution steels containing copper were amalgamated with mercury while metal substantially free from copper retained its characteristic grey surface. It is inferred that copper passes into solution with the iron ions, is plated back, and forms a mirror surface with reduced mercury. The passivity of iron in solutions of an alkali bichromate is partially or wholly destroyed in the presence of a copper salt, and it was found that 4—6 times more chromic acid per unit volume is required to prevent corrosion of a copper-bearing steel than is needed to protect iron free from copper. Galvanised sheets immersed in a 10 or 20% solution of aluminium sulphate corrode rapidly, hydrogen being liberated and the zinc coating stripped off. The reaction speed is then reduced greatly if the metal is pure iron, but with copper steels the reaction continues until the iron is dissolved entirely. The system usually turns to a hydroxide gel. A copper steel lost 5 oz. per sq. ft. in a 30-hr. test in boiling aluminium sulphate solution as compared with 2.5 oz. loss for a commercial pure iron. Results from acid tests are not definite, as such tests are sensitive to many electrochemical variables. Dilute sulphuric acid acted rather more vigorously on low copper steel (0.038% Cu) than on steel containing 0.20—0.25% Cu. The order of corrosion was reversed in hydrochloric acid.—C. A. K.

*Iron and steel; Corrosion of —.* J. A. Aupperle and D. M. Strickland. Trans. Amer. Electrochem. Soc., 1921, 167—177. [Advance copy.]

THE resistance to corrosion of varying grades of pickled iron and steel was determined by suspending plates (6×6 in.) in a solution containing 0.7% of sulphuric acid, 1.98% of ferrous sulphate, and 0.15% of ferric sulphate. The corroding solution was circulated and aerated continuously and was maintained at constant acidity. The loss in weight

in 60 days was as follows:—Pure open-hearth iron 0.622 oz. per sq. ft., copper open-hearth steel (0.257% Cu) 0.75, open-hearth steel 0.779, copper Bessemer steel (0.224% Cu) 2.060, copper acid open-hearth steel (0.209% Cu) 3.263. Metallic copper in contact with various kinds of iron and steel had little effect upon the corrosion of any of the metals with the exception of copper-bearing steel, which was attacked in areas surrounding the contact copper.—C. A. K.

*Corrosion of iron by acids; Effect of copper and silver salts on —.* O. P. Watts and H. C. Knapp. Trans. Amer. Electrochem. Soc., 1921, 135–140. [Advance copy.]

Corrosion of iron by sulphuric acid is increased in the presence of copper or silver salts in solution. In  $N/1H_2SO_4$  containing 2.5 g. Cu per l., corrosion due to voltaic action on copper steel (about 0.25% Cu) was nearly four times the direct corrosion by the acid, and in acid solution containing 0.1 g. Cu per l. the ratio of voltaic to direct corrosion was 0.85. Loss by voltaic corrosion of mild steel was of the same order, but the ratio (0.01) appears low owing to the great loss by direct acid corrosion. Figures obtained in tests in which silver salts were added to  $N/1$  sulphuric acid indicate less action by a large quantity of silver (2.5 g. per l.) than by either of the lesser quantities (0.1 and 0.01 g. Ag per l.). This is due to the formation of a deposit of spongy silver which retards diffusion of the acid to the steel. In  $N/50$  sulphuric acid voltaic action on copper steels and Armco iron appears to be 1–3 times the amount of corrosion due to the acid alone, but wide differences were observed in duplicate experiments. The serious effect of voltaic action is shown by the destruction of a monel metal yacht in which the steel parts in contact with monel metal were corroded away in three months.—C. A. K.

*Tin and zinc; Recrystallisation of —.* Recrystallisation of cold-worked metals. G. Masing. Int. Zeits. Metallg., 1920, 12, 457–493. Chem. Zentr., 1921, 92, II., 700–701.

THE surface tension theory of the recrystallisation of worked metals not only presents theoretical difficulties but also does not explain all the observed facts, e.g., the formation of nuclei, the growth of small crystals at the expense of their larger neighbours, and the failure of unworked metal to recrystallise. Czochralski's stratification theory (Int. Zeits. Metallg., 1916, 8, 1) satisfactorily explains these phenomena but at the same time it gives rise to a new series of problems, such as the nature, stability, and inner structure of a stratified crystal. The recrystallisation of cold-rolled tin begins with the formation of primary grains, the size of which depends on the recrystallisation temperature, and which, after at time, commence to quite, but more slowly than they form. The formation of nuclei cannot be observed, the nuclei of the primary grains being already present in the rolled tin. The size of the grains depends, according to Czochralski's theory, on the amount of rolling that the metal has undergone as well as on the recrystallisation temperature. If a worked piece of tin is subjected to a smaller secondary deformation of a different kind to the first (e.g. shearing after rolling, or rolling after hammering) a rapid formation of specially large crystals takes place as the result of two different stratifications. It only issues if the metal has originally been subjected to stronger primary deformation and is caused by the rapid grain-growth and by the formation of nuclei, which becomes quicker with rising temperature and with increase of secondary deformation, but tends to be reduced by the effect of the preceding primary deformation. The size of the

secondary crystals is in inverse proportion to the number of nuclei formed and the resulting metal is not changed immediately to an even granular structure but exhibits a series of recrystallisation steps with different but characteristic remains of stratification in the grains. Cold-rolled zinc shows a secondary formation of nuclei at 150° C. without being subjected to a secondary deformation; after this, however, recrystallisation commences at 70° C. as the metal is then in a specially unstable condition, which on continued working may again be changed to the normal state, so that the ordinary primary recrystallisation sets in.—A. R. P.

*Aluminium utensils; Blackening of —.* J. Czochralski. Z. Metallg., 1920, 12, 430–443. Chem. Zentr., 1921, 92, II., 599.

BLACKENING occurs readily in aluminium utensils in which water is kept warm. It is due to the deposition of a precipitate containing iron, silicon, and sulphur in greater proportion than the metal itself. No blackening is produced normally by aqueous solutions of iron salts; the blackening which occurs when aluminium is treated with a concentrated solution of ferric chloride in strong hydrochloric acid is an etching effect. Blackening occurs when tap water is boiled in an aluminium vessel, and the effect reaches a maximum when all carbonic acid is expelled (20 mins.). The presence of free acids is inhibitory and the phenomenon is connected with the alkalinity of the water. The absence of blackening in domestic practice may be due to the alternations of acid and alkaline conditions in cooking operations. The process is electrolytic and takes place at the eutectic  $Al-FeAl_3$  precipitation occurring on the aluminium. It is dependent on the degree of alkalinity and the most pronounced effect was caused by lime water. Synthetic tap water free from iron and spring water behaved like ordinary tap water, while rain water and distilled water produced no effect.—H. J. H.

*Nickel; Ductile electrolytic —.* C. P. Madsen. Trans. Amer. Electrochem. Soc., 1921, 239–276. [Advance copy.]

THE most satisfactory electrolyte for the production of thick uniform deposits of nickel was found to be nickel sulphate solution containing a small amount of alkali sulphate, a little boric acid and some nickel chloride. Cold-rolled malleable nickel anodes of the composition 95.3% Ni, 2.13% Fe, 0.25% Cu, 0.33% Mn, 0.30% C, and 0.23% Si, gave the best results in this electrolyte when the area of the anode was 1.77 times that of the cathode, the current density 72 amps. per sq. ft. of anode, and the temperature of the bath 55° C. If the cathodes were periodically lifted out of the bath, exposed to the air for a short time, and again immersed in the bath within a certain limiting time, the resulting nickel had a very great ductility, tensile strength, and hardness compared with ordinary nickel. The tensile strength of the deposited metal was about 72,000 lb. per sq. in.; after drawing down to wire of 0.002 in. diam. the tensile strength had increased to 250,000 lb., while the hardness increased from 130 to 150. The metal contained 99.7% Ni, 0.01% Fe, and 0.02% Cu. A tendency of the deposit to form pits was overcome by altering the composition of the anode.—A. R. P.

*Nickel deposition; Use of fluorides in solutions for —.* W. Blum. Trans. Amer. Electrochem. Soc., 1921, 227–247. [Advance copy.]

THE use of fluorides in conjunction with boric acid in nickel plating baths produces nickel deposits that have a finer texture and a higher tensile strength and hardness than those produced in chloride solutions, but the deposit usually contains more iron and copper. The following two solutions

are recommended as electrolytes: 255 g. of nickel sulphate, 12 g. of nickel carbonate, 8 g. of 50% hydrofluoric acid, and 31 g. of boric acid dissolved in water and made up to a litre; or 281 g. of nickel sulphate, 8.4 g. of sodium fluoride, and 31 g. of boric acid dissolved in water and made up to 1 litre. Adherent deposits for electroplating are obtained by using a current density of 3–4 amps. per sq. dm., while 1.5 to 2 amps. is sufficient for electrotyping. Methods for the analysis of the baths are discussed. —A. R. P.

*Chrome-nickel heating wires; Structure of —.* M. von Schwarz. Z. Metallk., 1921, 13, 125–127.

SATISFACTORY chrome-nickel wires for electrical heating purposes show a fine-grained structure both before and after prolonged use. The used wires show superficial oxidation and some slag inclusions below the surface. Inferior wires which have appeared on the market within the last few years have a similar fine-grained structure before use, but after use the structure becomes coarser and the wires become very brittle. Good wires contain about 86.5% Ni, 10.1% Cr, and only small quantities of iron, whilst a sample of the new inferior wire contained 52.7% Ni, 10.7% Cr, and 32.3% Fe. The inferiority of the latter is due to the replacement of so large a proportion of the nickel by iron. —E. H. R.

*Smelting; Theory of —. II. W. Guertler. Equilibria between pairs of metals and sulphur. I. The system copper-lead-sulphur.* K. L. Meissner. Metall und Erz, 1921, 18, 145–152.

GUERTLER (Metall u. Erz, 1920, 17, 192) has shown that when sulphur is added to a mixture of copper and lead,  $\text{Cu}_2\text{S}$  is first formed, then  $\text{PbS}$ , and finally  $\text{CuS}$ , and these relations were illustrated by means of a triangular diagram. In this diagram the ternary system is divided into three partial systems,  $\text{Pb-Cu-Cu}_2\text{S}$ ,  $\text{Pb-Cu}_2\text{S-PbS}$ , and  $\text{Cu}_2\text{S-PbS-S}$ . Various mixtures corresponding with points in each of these partial systems have now been examined thermally and microscopically. Each pair of constituents in the first ternary system shows incomplete miscibility, and consequently an area of incomplete miscibility would be expected in the ternary system, with three separate phases. Actually only two layers are formed, the upper consisting essentially of  $\text{Cu}_2\text{S}$ , the lower being an emulsion of lead and copper, in which the lead shows a pronounced tendency to liquefy downwards. Through this emulsion formation, copper and lead can be made practically completely miscible by addition of 1–1.5% of sulphur. Addition of lead to the  $\text{Cu-Cu}_2\text{S}$  system extends the miscibility gap. In the partial system  $\text{Pb-Cu}_2\text{S-PbS}$ , a miscibility gap extends almost up to the  $\text{Cu}_2\text{S-PbS}$  line. In the absence of free lead, i.e., on the  $\text{Cu}_2\text{S-PbS}$  line, only eutectic mixtures are formed. In the third ternary partial system,  $\text{Cu}_2\text{S-PbS-S}$ , it was expected that the line  $\text{PbS-CuS}$  would correspond with a quasi-binary series of mixtures, hnt, owing to the decomposition of  $\text{CuS}$  into  $\text{Cu}_2\text{S}$  and sulphur on melting, equilibrium was established between  $\text{PbS}$ ,  $\text{Cu}_2\text{S}$ , and an apparently ternary crystalline phase. —E. H. R.

*Alloys; Use of shear tests for judging the mechanical properties of —.* R. Krulla. Z. Metallk., 1921, 13, 137–139.

A SHEAR test is made on a test-piece having a cross-section of 0.5 sq. cm., and from the area of the shear diagram, measured by means of a planimeter, the shearing work is determined in kg.-m. per sq. cm. This quantity, divided by the Brinell hardness (previously determined on the same test-piece) gives a number  $Q$ , which serves to indicate the relation of strength to hardness in the alloy. Thus for

copper,  $Q=4.96$ , for the brass  $\text{Cu } 69.5\%$ ,  $\text{Zn } 30.5\%$ ,  $Q=8.34$ , and for  $\text{Cu } 49\%$ ,  $\text{Zn } 51\%$ ,  $Q=0.85$ .

—E. H. R.

*Mercury; Physical properties of —.* A. Schulze. Z. Metallk., 1921, 13, 105–113, 139–148.

A REVIEW of the literature dealing with the determination of the magnitudes of the physical properties of mercury, grouped under the headings mechanical, thermal, electrical and magnetic, and optical. —E. H. R.

*Reduction by metals in acid solutions.* Sugden. See VII.

#### PATENTS.

*Pig iron from iron and steel scrap; Production of high-grade —.* F. Rottmann. G.P. 332,208, 19.3.19.

CLEAN iron and steel scrap is melted with a pure slag made from quartz, lime, or clay shale. Iron low in phosphorus may thus be obtained. —H. J. H.

*Iron and steel baths; Preventing the re-phosphorizing of — during deoxidation and carburising.* Deutsch-Luxemburgische Bergwerks- und Hütten-A.-G. G.P. 333,749, 19.4.16.

THE bath of molten iron or steel is treated with calcium carbide, at or towards the end of the deoxidation or carburising operations. The method is especially applicable to the Thomas process where the slag is high in phosphorus, and to the manufacture of hard steel. —A. R. P.

*Ferro-chromium; Process for producing low-carbon —.* F. Krupp A.-G. E.P. 145,710, 26.6.20. Conv., 3.10.16. Addn. to 145,709 (J., 1921, 307 A).

CRUDE ferro-chromium is treated in an acid-lined converter until a low carbon content is obtained. Basic material, e.g. lime, is then added to the charge, whereby silicon is oxidised and the loss of chromium is reduced. —O. A. K.

*Ferrotitanium; Process for producing low-carbon —.* H. C. Sicard, Assr. to U.S. Ferro Alloys Corp. U.S.P. (A) 1,374,035 and (b) 1,374,036, 5.4.21. Appl., 18.8.19.

(A) COMMERCIAL ferrotitanium is mixed with an excess of titanic oxide and heated in an electric furnace so as to decompose carbides of titanium and iron and form a protective titanium oxide slag. (b) A double carbide of titanium and iron is treated in a molten state with aluminium. Carbon separates as graphite and is skimmed off, and the underlying metal is reheated and cast. —C. A. K.

*Ferro-zirconium; Production of low-carbon —. Process for producing zirconium steel.* H. C. Sicard. U.S.P. (A) 1,374,037 and (b) 1,374,038, 5.4.21. Appl., 23.7.20.

(A) A HIGH-CARBON ferro-zirconium alloy is decarbonised by means of titanic oxide. (b) An alloy of zirconium and silicon is added to molten steel. —B. M. V.

*Roasting and sintering ores and the like.* R. L. Lloyd. E.P. 160,477, 4.9.19.

AN improved machine for carrying out mechanically the operations of charging, igniting, sintering, and discharging in the Dwight-Lloyd process, as described in E.P. 25,985 of 1907 (J., 1908, 1025). —B. M. V.

*Solid substances capable of reaction with a gaseous reagent; Process of treating —.* [Roasting sulphide ores.] E. C. R. Marks. From Ore Roasting Development Co. E.P. 161,103, 6.7.20.

A MULTIPLE-HEARTH mechanical roaster is provided with by-pass ore passages, so that a portion of the

ore can fall direct from the second or any subsequent hearth to some lower hearth. One or more by-passes may be provided, so that after initial heating on the top hearths the ore will divide into two or more streams which will subsequently be united into one before final discharge from the apparatus. Air, or other gas, may also be admitted at several different places, preferably near the outlets of the by-passes, and some of the products of combustion may be readmitted.—B. M. V.

*Electrode for use in electric welding or fusion deposition of metals.* Quasi Arc Co., Ltd., and A. P. Strommenger. E.P. 160,957, 7.1.20.

An electrode wire is wrapped spirally with a yarn of blue asbestos. The asbestos fibres are pulped with a solution of sodium silicate and rolled into a thin sheet, which is cut into strips and spun into a spirally twisted yarn.—C. A. K.

*Slag; Process of cleaning converter* —. H. H. Stout, Assr. to Phelps Dodge Corp. U.S.P. 1,372,047, 22.3.21. Appl., 3.2.19.

For the removal of copper from converter slag the specific gravity and silica content of the latter are adjusted to those of the reverberatory slag before the two are mixed.—D. F. T.

*Electrolytic recovery of metals from solutions; Electrode for* —. U. C. Tainton. U.S.P. 1,373,273, 23.3.21. Appl., 4.11.19.

A PERMEABLE electrode suitable for the electrolytic deposition of metals from solutions is composed of a paper sheet in which is incorporated a comminuted conducting material.—J. S. G. T.

*Electrolytic process [for treating copper ores].* W. E. Greenawalt. U.S.P. 1,373,557, 5.4.21. Appl., 22.3.20.

COPPER ores are treated with an acid solution, and copper is deposited electrolytically from the resulting solution containing also salts of elements of variable valency (iron etc.). These latter salts are reduced from the higher to the lower valency by agitating the electrolyte in the presence of sulphur dioxide and finely divided charcoal.—J. S. G. T.

*Furnace; Revolving* —. L. P. Basset. U.S.P. 1,372,392, 22.3.21. Appl., 12.9.19.

An inclined rotating cylindrical furnace has its diameter increased at the lower end to form a reaction chamber adapted to form a pocket for molten metal.—B. M. V.

*Furnace [; Open-hearth* —]. N. F. Egler, Assr. to Blair Engineering Co. U.S.P. 1,372,613, 22.3.21. Appl., 1.3.19.

MEANS are provided whereby air may be passed from the regenerators to combined air and fuel ports at each end of the furnace, which are narrow compared with the hearth, and also through other channels direct to the interior of the furnace; the latter channels may be shut off if desired.—B. M. V.

*Melting furnace [; Electric brass* —]. T. F. Baily and F. T. Cope, Assrs. to The Electric Furnace Co. U.S.P. 1,373,787, 5.4.21. Appl., 16.2.20.

An electric furnace for melting brases, in which the heat is produced by the resistance of an annular trough of coarsely powdered carbon, is fitted with a condenser connected with the upper part of the heating chamber and provided with an inclined neck communicating with the heating chamber near the hearth of the furnace.—A. R. P.

*Blast furnaces; Process for operating* — with the use of lignite. F. Weeren. G.P. 331,596, 11.1.12.

A LARGE proportion of the coke or charcoal in the charge is replaced by lignite briquettes, and the ore

and limestone are added in small lumps or as a powder. In the upper part of the furnace the bituminous constituents of the lignite reduce the iron oxide in the ore, and the carbonised briquettes break up into small pieces which serve for smelting and carburising the iron.—L. A. C.

*Copper; Process of case hardening* —. A. S. Gundersen. U.S.P. 1,372,423, 22.3.21. Appl., 1.12.15.

THE surface of the copper is exposed to zinc at a temperature between the melting points of copper and zinc.—B. M. V.

*[Aluminium] ores; Process for the reduction of* —. C. G. Collins, Assr. to C. A. Stevens. U.S.P. 1,372,483, 23.3.21. Appl., 26.3.20.

THE finely ground ore is mixed with a carbonaceous material and sodium chloride, and the mixture is heated out of contact with the air.—A. R. P.

*Aluminium; Process for coating metallic objects with* —. Metallhütte Baer und Co. Kommanditges. G.P. 330,917, 11.4.19.

FOR the coating of objects made of metals harder than aluminium a mixture of hard sand with powdered aluminium, in which the particles of the latter are larger than those of the former, is projected against the surface of the object by means of air under a pressure of 1–4 atm.—J. H. L.

*Aluminium; Electrolytic manufacture of* —. Allgem. Elektrizitäts-Ges. G.P. 332,669, 1.3.18.

ALTERNATING current is passed through the bath for the purpose of heating, without any heating resistances, whilst at the same time electrolysis is effected by a direct current. The danger of the solidification of the bath is thus avoided in case of defects at the generator of the direct current, and losses in the transformation of the generally available alternating current into direct current are minimised.—C. A. C.

*Precious metals [gold and silver]; Method of recovering* — from solutions. R. H. McKee. U.S.P. 1,372,971, 29.3.21. Appl., 13.5.20.

SOLUTIONS containing gold and silver are brought into contact with carbon which has previously been subjected to an alkaline treatment at a suitable temperature and subsequently leached before use.—A. R. P.

*Silver; Method of extracting* — [from ores or residues]. P. R. Middleton, Assr. to J. C. Lalor. U.S.P. 1,372,973, 29.3.21. Appl., 11.5.20.

ORES or residues containing metallic silver are subjected to treatment to convert the silver into sulphide, which is then chlorinated.—A. R. P.

*Zinc and cadmium; Process for separating and recovering* — by acid treatment. J. Leibn. G.P. 329,172, 9.6.17. Addn. to 322,142 (J., 1920, 788A).

THE residues containing cadmium are heated before treatment with acid. The formation of malodorous substances is thereby avoided.—J. H. L.

*Metals; Process for recovering* — from ores. H. Dahlem. G.P. 332,445, 4.2.20. Addn. to 330,290 (J., 1921, 265A).

IN the process described in the chief patent the furnace is so designed that air or gas for reduction can be blown independently into the furnace through all the tuyères, both at the sides and at the bottom of the furnace.—L. A. C.

*Metallic coatings; Production of* — by spraying atomised liquid metals. P. Künzler. G.P. 330,916, 20.9.13.

THE metal is melted for the spraying operation by



conducting it, in a comminuted but not pulverulent form, through a duct which is strongly heated from without. By this means metals which are difficult to melt and readily oxidisable, such as aluminium and copper, can be used in the spray coating process.—J. H. L.

*Coating articles by spraying with a dispersed liquid material.* P. Künzler. G.P. 332,047, 21.10.13. Addn. to 330,916 (*cf. supra*).

The coating material is used in form of pieces of regular shape, especially balls. The method is of special value in the case of easily oxidisable metals, *e.g.*, aluminium, which might solidify and oxidise when fused in large quantities.—C. A. C.

*Etching and stamping processes; Plates for —.* Verein. Elektrochem. Fabr. O. Hahn. G.P. 330,948, 21.11.19.

The plates are made of magnesium or magnesium alloys; they are very hard and suitable for etching and engraving.—J. H. L.

*Sludge from wet purification of blast-furnace gas; Removal and utilisation of —.* F. Eckert. G.P. 332,628, 30.11.19.

By mixing the sludge with the slag from the blast-furnaces the water content of the sludge is evaporated by the heat of the slag, whilst the dry residue adheres to the granulated slag. The hydraulic properties of the slag are improved by the admixture of the sludge.—C. A. C.

*Steel; Manufacture of —.* A. Pacz, Assr. to General Electric Co. U.S.P. 1,373,908, 5.4.21. Appl., 17.8.17.

SEE E.P. 152,371 of 1918; J., 1920, 787 A.

*Alloys.* F. Milliken. E.P. 161,104, 6.7.20.

SEE U.S.P. 1,354,990 of 1920; J., 1920, 754 A.

*Welding of different metals to form a composite ingot.* A. G. C. Pitteril. U.S.P. 1,374,110, 5.4.21. Appl., 14.10.18.

SEE E.P. 122,365 of 1918; J., 1919, 183 A.

*Blast-furnace gas.* E.P. 153,263. See IIA.

*Tunnel furnace.* G.P. 331,703. See VIII.

## XI.—ELECTRO-CHEMISTRY.

*Electric furnaces; Electro-dynamic forces in —.* C. Hering. Trans. Amer. Electrochem. Soc., 1921, 201—211. [Advance copy.]

THE author discusses the nature of the mechanical forces which may be directed to cause an increased directional motion of a liquid conductor, *e.g.*, molten metal, in an electric furnace. The "pinch" effect (J., 1909, 1143) is now applied to many furnaces. The author reiterates his view that a component of the electro-dynamic force may act in an axial direction when two currents are inclined to each other. The rising of metal on the outer side of the ring of an induction furnace is explained as due to the "stretching" of the current-carrying bath of metal, and not indirectly as the result of hydraulic forces.—C. A. K.

*Fluorine.* Meyer and Sandow. See VII.

*Electric furnaces.* Mylius. See X.

*Chrome-nickel wires.* Von Schwarz. See X.

## PATENTS.

*Electric [arc] furnaces.* The British Thomson-Houston Co., Ltd., H. C. Hastings, and N. Laycock. E.P. 160,529, 19.12.19.

IN electric arc furnaces to which power is supplied from single-phase or two-phase four-wire circuits, and in which there are two arcs in series per phase, one of the arcs being controlled by the arc current and the other by the voltage across the arc as described in E.P. 119,228 (U.S.P. 1,255,613; J., 1918, 213 A) a system of magnetic relays operates to prevent the voltage-controlled electrode from being lowered when the current is abnormally high, and to prevent the current-controlled electrode from being lowered when the arc voltage is abnormally high. Relays are provided whereby one or both of the electrodes per phase may be raised at a higher speed than the normal operating speed during the period when the current in the arcs exceeds a predetermined value.—J. S. G. T.

*Storage battery electrode and process of making same.* H. C. Hubbell, Assr. to Hubbell-Fuller Battery Co. U.S.P. (A) 1,373,733 and (B) 1,373,734, 5.4.21. Appl., 9.7.15. Renewed 13.8.20.

(A) A storage battery electrode is made by heating nickel oxide with finely divided cadmium. (B) Precipitated cobaltous hydroxide is converted into oxide by heating it without substantial exposure to air. The oxide is cooled under the same conditions and mixed with a material containing nickel, and the mixture formed into tablets and assembled in a conducting container.—J. S. G. T.

*Storage battery plates; Process of making —.* W. H. Grimditch, Assr. to Philadelphia Storage Battery Co. U.S.P. 1,374,076, 5.4.21. Appl., 24.7.20.

LEAD oxide containing red lead is mixed in definite proportion with a solution of ammonium sulphate of a definite strength, at a predetermined temperature, such that the mixture has the plasticity required for applying it to the battery grid.

—J. S. G. T.

*Lead peroxide-zinc cells or batteries; Process of making — capable of being kept.* Ringe and Co. G.P. 333,299, 6.1.18.

SUBSTANCES such as the hydroxides of calcium, magnesium or barium, are added to the electrolyte or to the positive electrode, or to both, in order to prevent the formation of lead chloride.—A. R. P.

See also pages (A) 333, *Electrical precipitation* (U.S.P. 1,371,995); *Cleaning gases* (U.S.P. 1,372,710). 346, *Electrolysing vat* (U.S.P. 1,372,176); *Fixation of nitrogen* (U.S.P. 1,373,639). 353, *Electrode* (E.P. 160,957 and U.S.P. 1,373,273); *Electrolytic process* (U.S.P. 1,373,557); *Electric melting furnace* (U.S.P. 1,373,787). 362, *Sugar manufacture* (U.S.P. 1,371,997). 366, *Water purification* (E.P. 158,620). 368, *Acetaldehyde from acetylene* (E.P. 143,891).

## XII.—FATS; OILS; WAXES.

*Twitchell reagent; The —.* E. Hoyer. Z. Deuts. Oel- und Fettind., 1921, 41, 113—115. Chem. Zentr., 1921, 92, II., 665—666.

IN the Twitchell process, the aromatic sulpho-fatty acid serves to emulsify the fat or oil, whilst the free sulphuric acid present effects the hydrolysis; removal of the sulphuric acid by washing or treatment with normal sodium sulphate, sodium bisulphite, or sodium acetate retards the hydrolysis, as also does the addition of formic, acetic, or sulphurous acid. The Petroff reagent consisting of sulphonic acids obtained as a waste product in the purification of petroleum is considered superior to the Twitchell reagent. The discoloration which occurs during hydrolysis depends on the amount of



sulphuric acid present, so that it is advisable to use as little of this acid and of the reagent as possible; the resulting fatty acids are more susceptible to discoloration than are their parent fats and oils.

—D. F. T.

*Oil of Gillettiella congolana; Properties and composition of the —.* A. Boulay. Bull. Sci. Pharmacol., 1920, 27, 626–628. Chem. Zentr., 1921, 92, I., 576.

THE seeds of *G. congolana* collected in the neighbourhood of Matadi were at most 45 mm. long, and 25 g. in weight. The oil extracted by petroleum spirit is clear, but deposits crystals in the course of a few days. It has the following characters:—Sp. gr. 0.9159, free oleic acid 0.79%, saponif. value 192.5, iodine value 93.8, Hehner value 90, soluble volatile acids 0.77%, acetyl value 7.0, unsaponifiable matter 1.14%, m.p. of fatty acids 29°; the fatty acids consist of 35% of solid acids (palmitic etc.), and 65% of liquid acids (linolic and oleic acids).

—G. F. M.

*Fats; Law of probability applied to the formation of — from carbohydrates.* E. J. Witzemann. J. Phys. Chem., 1921, 25, 55–60.

IF the relative abundance of occurrence of the various fatty acids is plotted against the number of carbon atoms in the molecule, and the points corresponding to the relative abundance of the various acids are connected, instead of obtaining a smooth curve on both sides of  $C_{18}$ , the curve shows prominences at several points, and therefore does not conform with the typical probability curve. But, if the prominences are connected, such a curve is obtained. The numbers of carbon atoms at the prominences are all divisible by six, but  $C_{18}$  fatty acids occur considerably more frequently than others; this affords support for Fischer's hypothesis of the formation of fatty acids from sugars. The less frequent occurrence of intermediate acids may be due to their formation from higher and unsaturated acids, or by synthesis from short carbon chains.

—J. C. K.

*Catalytic activity of platinum; Influence of mercury, sulphur, arsenic, and zinc on the —.* E. B. Maxted. Chem. Soc. Trans., 1921, 119, 225–233. (Cf. J., 1921, 89 A.)

THE inhibitive action of mercury, sulphur, arsenic, and zinc on the catalytic activity of platinum when employed for the catalytic hydrogenation of oleic acid under standard conditions was studied by similar means to those previously employed in the case of inhibition by lead (*loc. cit.*), and the existence of a similar linear poisoning law has been established. For the above substances the linear law appears to hold between the limits of zero concentration of inhibitor up to a region in the neighbourhood of total extinction, where a point of inflexion occurs from which the poisoning curve slopes far less steeply towards complete inactivity.—G. F. M.

*Herabromide value of fatty oils; Determination of —.* A. Eibner. Farben-Zeit., 1921, 26, 1314–1315.

THE method of Bailey and Baldisiefen (J., 1921, 51 A) gives lower values than those obtained by the methods of Eibner and Muggenthaler (J., 1913, 242), Wolff (J., 1920, 417 A), and Steele and Washburn (J., 1920, 197 A). The presence of fish oils as adulterants of linseed oil may be detected by testing the solubility of the octobromides in boiling benzol and determination of the melting point of the solute. A further useful method of recognition of fish oils is that of Tsujimoto (J., 1920, 825 A) depending on the separation of clupanodonic acid as the lithium salt.—A. de W.

*Partly hydrolysed fats (soap-stock); Analysis of —.* W. Fahrion. Chem. Umschau, 1921, 28, 68–69.

THE aqueous layer from the alkali refining of vegetable oils yields on acidification a product which contains neutral fat in addition to fatty acids, and is largely used in soap manufacture. Errors may occur when the free fatty acid content of the material is calculated from the ratio of the acid value to the saponification value, since the equivalent weight of fatty acid is less than that of its corresponding glyceride by the equivalent weight of the glyceryl radicle. Error is also caused by the small differences existing between neutralisation and saponification values of free fatty acids. In many cases, however, these errors compensate one another sufficiently for all practical purposes. The author recommends the following procedure: A weighed quantity of the soap stock is warmed with alcohol and the acid value determined. A measured amount of alcoholic alkali is then added and the ester value determined in the usual way. The neutral soap is then rendered alkaline, diluted to an alcoholic strength of 50–20%, and the unsaponifiable matter (y) extracted with petroleum-spirit, or ether. The free fatty acid (x) then corresponds to  $(100-y) \times \text{acid value/saponif. value}$ , and the percentage of neutral fat is obtained from the difference:  $100-(x+y)$ .—A. de W.

*Oxidation of paraffin wax.* Fischer and Schneider. See IIA.

*Vitamin in fats.* (1) Hopkins. (2) Drummond and Coward. (3) Zilva. See XIXA.

*Lard.* Drummond and others. See XIXA.

#### PATENTS.

*Fats and oils; Process for the removal of fatty acids, resins, bitter and mucilaginous substances from —.* H. Bollmann. U.S.P. 1,371,342, 15.3.21. Appl., 22.6.20.

A CONTINUOUS current of diluted solvent for the impurities is passed upward through a systematic leaching apparatus comprising a number of vessels each containing a body of stationary filling material, whilst a continuous current of the oil to be purified passes through in a downward direction, whereby intimate contact of oil and solvent is produced without violent agitation of the materials, and the quantity of difficultly separable emulsion formed is reduced to a minimum.—A. de W.

*[Vegetable] oils; Process of treating [purifying] —.* Treatment of castor oil. A. Schwarzman, Assr. to S. Kellogg and Sons, Inc. U.S.P. (A) 1,372,631 and (B) 1,372,632, 22.3.21. Appl., 19.2. and 20.4.20.

(A) VEGETABLE oils are agitated with an amount of strong caustic soda solution corresponding to the content of free fatty acids in the oil, the temperature is raised to about 180° F. (82° C.), and hot water passed, in finely divided form, into and through the mixture. (B) Castor oil is mixed with a limited amount of soda solution of moderate strength to neutralise the free fatty acids, hot water added with thorough agitation, the aqueous layer allowed to settle, the oil washed with hot water, and finally dried.—A. de W.

*Fatty acids; Distillation of —.* J. W. Bodman, Assr. to W. Garrigue and Co., Inc. U.S.P. 1,372,477, 22.3.21. Appl., 9.8.19.

To remove their dark colour fatty acids are brought, in the form of spray only, into contact with a gaseous carrier, in a vacuum, under heat conditions suitable for the complete distillation of the acids. The evaporated fatty acids are condensed.—A. de W.

*Fatty matter; Extraction of — from garbage and other fat-containing materials.* V. Bredlik and L. C. Whiton, jun. U.S.P. 1,372,479, 22.8.21. Appl., 15.5.19.

IN extracting the fatty matter from garbage and other material containing grease or oil by means of a solvent, the material to be extracted is subjected to a preliminary treatment of alternately higher and lower gaseous pressures in the absence of the solvent.—A. de W.

*Fatty acids and their glycerides; Production of —.* Oelverwertung G.m.b.H. G.P. 330,811, 22.8.12. Addn. to 292,649.

A MIXTURE of nickel oxide with fat or a fatty acid is heated in a current of hydrogen, and the colloidal product is used as a catalyst in the hydrogenation of oils. By this means only a small proportion of the oil need be heated to the high temperature necessary for the colloidal distribution of the catalyst. A hard fat with a setting pt. about 50° C. may thus be prepared from cottonseed oil, and dihydroxystearic glycerol ester from castor oil.

—J. H. L.

*Saponaceous compositions; Manufacture of —.* F. G. Chadbourne. E.P. 180,892, 1.12.19.

FATTY acids are melted in a steam-jacketed pan and incorporated in a rotary mixer with 20–50% by weight (on the final product) of a finely divided hydrated aluminium silicate, e.g., china clay, together with colouring matter. The required quantity of alkaline solution is then run in, the mixing operation continued for about 1 hr. at a temperature below boiling-point, steam being admitted through a steam coil, and the mass is concentrated to the desired consistency.—A. de W.

*Emulsion and method of producing it.* E. M. Johansen, Assr. to The Atlantic Refining Co. U.S.P. 1,373,661, 5.4.21. Appl., 14.12.20.

MINERAL oils are emulsified with water in the presence of water-soluble sulphonates, obtained by the acid treatment of mineral oils or products therefrom, to the extent of about 1.5 to 8% of the weight of mineral oil.—L. A. C.

*Detergent.* Chem. Fahr. vorm Weiler-ter Moer. G.P. 330,355, 24.2.17. Addn. to 327,683 (J., 1921, 186 A).

INSTEAD of pulverulent insoluble substance a mixture of soluble salts is used which gives rise to a fine precipitate when introduced into water, e.g., xylene mixed with magnesium sulphate and sodium carbonate and sulphate, or chlorobenzene mixed with aluminium sulphate and sodium carbonate.

—J. H. L.

*Detergent which lathers well; Production of a —.* E. Reinfurth. G.P. 332,649, 1.8.16.

SOLUBLE salts of  $\alpha$ - or  $\beta$ -naphthalenemonosulphonic acid, or of both  $\alpha$  and  $\beta$  acids, preferably alkali or ammonium salts, are added to a soap during or after the production of the latter, and a filling agent, such as clay, may also be added. As much as 50–70% of the fatty acid may be replaced by the above-mentioned acids without any loss of lathering power.—J. H. L.

*Detergent and emulsive agents; Manufacture of —.* R. Macpherson and W. E. Heyes. U.S.P. 1,373,900, 5.4.21. Appl., 23.5.16.

SEE E.P. 8478 of 1915; J., 1916, 852.

See also pages (A) 333, *Soap powder* (E.P. 145,048). 339, *Mountain wax* (G.P. 334,155). 358, *Moulded article from tung oil* (U.S.P. 1,372,114). 365, *Culinary product. Edible oil* (U.S.P. 1,372,614–5); *Maize meal* (G.P. 328,425).

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Ferric oxide; Anhydrous yellow —.* J. H. Yoe. J. Phys. Chem., 1921, 25, 196–200.

IN an extension of Scheetz's work on the stabilisation of the colour of anhydrous yellow ferric oxide (J., 1917, 1270), aqueous solutions of ferrons and aluminium sulphates were precipitated with calcium and barium hydroxides, and the precipitate heated to 1000° C.; a yellow or buff colour persisted in mixtures containing up to about 8% of ferric oxide, but above this amount the colour gradually darkened. Similar results obtained with ferrous and aluminium chlorides indicate that the presence of calcium or barium sulphate in the mixture is not essential for stabilisation, but that alumina alone is sufficient to prevent agglomeration and to maintain the yellow colour of the ferric oxide. This conclusion is confirmed by precipitating both sulphates and chlorides of iron and aluminium with ammonia solution. In regard to calcium and barium sulphates, it is found that if these are present in large excess they act as stabilising agents; thus a product containing 6% of ferric oxide and 94% of barium sulphate is of a light buff colour similar to that obtained in presence of alumina. Attempts to retain the colour in products stabilised with alumina after removal of the latter by sodium hydroxide were unsuccessful, as agglomeration of the ferric oxide with corresponding change of colour to red, occurred in each case.—W. J. W.

*Paint films; [The "Paint-Film Gauge," an] instrument for measuring the thickness of wet —.* A. H. Pfund. J. Franklin Inst., 1921, 191, 517–523.

THE instrument consists of a plano-convex lens of 25 cm. radius of curvature mounted in a short tube sliding freely in an outer tube. The two tubes are held together by springs, and by applying pressure the inner tube can be forced outwards and the convex surface of the lens brought into contact with e.g. a wet paint film. The diameter (D) of the circular spot of wet paint left on the lens when the pressure is released is measured to 0.1 mm., and the thickness (t) of the paint film can then be calculated by the formula:  $t = D^2/16R$ , where R is the radius of curvature of the lens. Good agreement was shown in practice between results obtained by the instrument and results of actual spreading trials with paints and varnishes. Shrinkage of paint films on drying may be determined by means of the instrument, the thickness of the dry film being measured with a micrometer. The variation in brightness of paint films on drying may be measured by determining the thickness of the film at the point of critical opacity in a wedge-shaped pool of the paint, the thickness at several points at various other distances from the apex of the wedge being also determined. After drying, the change in position towards or away from the wedge apex of the point of critical opacity is observed and the thickness of the wet paint film formerly overlying this spot is obtained from the previous measurements. It was found that opacity in paints does not vary with shrinkage but increases directly as the brightness.—A. de W.

*Ink; Acidity of — and the influence of bottle glass on ink.* C. A. Mitchell. Analyst, 1921, 46, 129–134.

THE stability of iron-gall inks depends chiefly on the amount of acid present, and this amount must be kept within definite limits; the acid usually added is hydrochloric acid. Certain inks, consisting essentially of iron gallate solution, do not re-

quire the addition of mineral acid, but it is held generally that these inks lack "body." To determine mineral acid in ink, a portion of the sample is distilled with the addition of sodium acetate until the whole of the liberated acetic acid has passed over into the distillate; the acidity of the latter is then titrated. The total acidity of the ink is determined by boiling 5 c.c. of the sample with 10 c.c. of hydrogen peroxide under a reflux condenser until the colour has been discharged and then titrating the cooled mixture with sodium hydroxide solution, using phenolphthalein as indicator. Log-wood inks require to be boiled with alkaline hydrogen peroxide solution in order to destroy the colour; an allowance is made for the amount of alkali added. Results of determinations of acidity of commercial inks are recorded; usually the total acidity is equivalent to 1.8–5.6 c.c. of *N*/1 alkali solution per 10 c.c. of ink, and the mineral acidity to 0.3–2.46 c.c. of *N*/10 alkali solution per 10 c.c. Investigation of certain deteriorated inks showed that the damage had been caused by alkali dissolved from the glass bottles.—W. P. S.

*Pine resin; Acid constituents of* —. *d- and l-pimaric acids.* G. Dupont. *Comptes rend.*, 1921, 172, 923–925.

By the use of aqueous alcohol as solvent it has been found possible to isolate appreciable quantities of *l*-pimaric acid from the resin of the leaf buds of *Pinus maritima*. This acid is readily isomerised to the dextro-acid under the conditions usually employed for the extraction of pimaric acid from this source.—W. G.

*Rosin; Action of trichloroethylene on* —. F. Goldschmidt and G. Weiss. *Z. Deuts. Oel- und Fettind.*, 1921, 41, 99–100. *Chem. Zentr.*, 1921, 92, II., 651.

A black deposit in the salting out of soaps prepared from a mixture of equal parts of stearin and rosin which had been extracted with trichloroethylene was traced to the presence of less volatile impurities in this solvent. Rosin dissolved in an unsaturated compound such as turpentine or allyl alcohol yields dark-coloured products when treated with air in the presence of iron.—D. F. T.

*Furfural; Some synthetic resins from* —. G. H. Mains and M. Phillips. *Chem. and Met. Eng.*, 1921, 24, 661–663.

The condensation of furfural, which can now be produced very cheaply on a large scale from maize cobs, with various amines and ketones has been studied and the optimum conditions for the production of fusible resins suitable for use in varnishes are given. One part of furfural heated with an equal weight of aniline for 1 hr. at 200° C. or with twice its weight of  $\alpha$ -naphthylamine for 3 hrs. at 200° C. gives a black resin which is hard and brittle at 25° C. In some cases addition of hydrochloric acid to the reaction mixture is necessary; thus one pt. of furfural condenses with 2 pts. of *o*-toluidine or with 1 pt. of crude xylydine only in the presence of 0.2 pt. of strong hydrochloric acid at 150° C. to yield a hard black resin. Condensation products with acetone or methyl ethyl ketone are obtained on heating suitable mixtures in the presence of 50% sodium hydroxide solution. A black resin is obtained by heating furfural with 25% caustic soda for 1 hr. and treating the solution with hydrochloric acid. All the resins obtained softened between 25° and 100° C. were practically insoluble in water, somewhat soluble in turpentine, readily soluble in benzene, acetone, and alcohol, and very soluble in furfural. Solutions of the resins in benzene, acetone, or furfural give shades on wood ranging from golden brown to black, according to the nature and concentration of the solution.—A. R. P.

*Varnishes; Testing of* —. H. Wolff. *Farben-Zeit.*, 1921, 26, 1186–1187.

Films of the various varnishes under examination dried under similar conditions of time, etc., on both glass plates and tinned iron plates, are hung up in large glass bell-jars filled with a mixture of moist sulphur dioxide and carbon dioxide for a period of from 8 days to 8 weeks. Other tests comprise leading the mixed gases, both at ordinary temperatures and heated by passage through a tube immersed in a boiling water-bath, over the surfaces of the films (locomotive varnish test). Criteria as to the relative resistance of the varnishes can then be obtained by macroscopical and microscopical inspection, rubbing with the finger, etc.

—A. de W.

*Lead, manganese, and cobalt as driers.* W. Flatt. *Farben-Zeit.*, 1921, 26, 1441.

The relative speed of drying at 10° C. of oils boiled with various driers was found to be as follows: Lead drier (1 pt. Pb in 100 pts. of oil), 20 hrs.; manganese (1 in 400), 20 hrs.; cobalt (1 pt. 1200), 16 hrs. The form in which the base is dissolved in the oil is without influence on the drying power. A boiled oil containing 1% Pb is, however, extraordinarily heavy-bodied, and eventually becomes turbid and deposits "foots" possessing the same siccative power as the soluble part of the lead. The speed of drying at different temperatures of boiled oils varies with the nature of the drier; thus, exposure at 5° C. of the boiled oils referred to above in the order named, results in dry films being obtained in 24, 44, and 36 hrs. respectively. The progress of oxidation is dissimilar with the three bases; thus with a lead drier the oil showed definite signs of oxidation (thickening etc.) in 12 hrs., with cobalt there were no signs of drying before 13 hrs., whilst with manganese the oil showed no alteration before 20 hrs. exposure. A mixture of oils boiled with a lead drier and a manganese drier respectively dried in 16 hrs., the component oils when tested separately drying in 20 hrs. and 24 hrs. respectively. In a similar manner a 20-hr. lead-boiled oil mixed with a 16-hr. cobalt-boiled oil dried in 16 hrs. Relatively small amounts of manganese produce considerable acceleration of drying in oils boiled with lead driers. Manganese-boiled oils increase in drying power on ageing to a degree equal to that obtainable by mixing with lead-boiled oils.

—A. de W.

*Pressure oxidation of phenols.* Fischer and Schrader. See III.

#### PATENTS.

*White lead; Apparatus for the manufacture of* —. E. W. Dahl. E.P. 160,395, 2.7.20.

The apparatus consists of two wooden boxes each provided with an endless belt enclosed in a casing. The material, e.g., comminuted lead, acetic or nitric acid, and water is transferred by one belt from the lower portion of one box to the upper portion of the second, where it is discharged and falls over a series of inclined baffle plates and on to the lower portion of the second belt, the operation being repeated continuously and the reacting material continually passing from one box to the other. The belts are carried on wooden rollers mounted on driving shafts. The boxes are provided with charging hoppers and discharging traps, man-holes, and means for the inlet and discharge of carbon dioxide, air, and steam.—A. de W.

*Drying chambers, more particularly for dye, varnish, lacquer, and enamel-coatings.* Standard Lack Werke, G.m.b.H. E.P. 147,562, 8.7.20. Conv., 21.10.13.

A closed drying chamber is provided with two heat-

ing coils on opposite sides, near the bottom, behind baffles, in order to induce an upward circulation of heated air. A cooling coil is set in the middle plane of the drying chamber close below the roofing and is provided with a trough to receive condensed water. A "transferring device" influenced by the humidity of the chamber acts upon a valve either to admit water to the cooling coil, thereby effecting a reduction of humidity in the chamber by causing condensation, or to allow cold water to pass directly to the open trough beneath the coil, thereby increasing the humidity.—A. de W.

*Phenolic bodies; Condensation of — with aldehydic compounds.* Vickers, Ltd., The Loco Proofing Co., Ltd., and W. H. Nuttall. E.P. 160,258, 15.12.19.

PHENOLIC substances and aldehydic compounds or compounds capable of producing formaldehyde are condensed by the aid of a catalyst comprising a salt or salts of hexamethylenetetramine, e.g., the chloride, sulphate, phthalate, and/or a double compound thereof with phenol, pyrogallol, ethyl bromide, potassium antimony tartrate, etc., the proportion of the catalyst being such that the quantity of hexamethylenetetramine does not exceed 2% of the weight of the main ingredients of the composition. The character of the product may be varied by altering the relative proportions of phenolic substance and aldehydic compound and the nature of the catalyst or mixture of catalysts.—A. de W.

*Moulded article [from tung oil] and method of making same.* L. H. Baekeland, Assr. to General Bakelite Co. U.S.P. 1,372,114, 22.3.21. Appl., 12.1.18. (Cf. U.S.P. 1,312,093; J., 1919, 730 A.)

TUNG oil is heated to a relatively high temperature short of that at which solidification occurs, the treated oil mixed with an inert filling material, the mixture moulded, baked, and an insoluble external coating applied.—A. de W.

*Resinous composition; Fusible odourless — and process of making same.* W. E. B. Baker and H. Weaver. U.S.P. 1,373,044, 29.3.21. Appl., 23.4.20.

A FUSED resin is caused to react with an oxidising compound adapted to liberate nascent oxygen and destroy the odoriferous constituent originally present in the resin without substantially affecting the properties of the main constituent of the resin.—A. de W.

*Fumigant paint.* H. A. Gardner. U.S.P. 1,373,499, 5.4.21. Appl., 27.12.20.

A FUMIGANT coating composition contains a chlorinated derivative of benzol. (Cf. J., 1921, 187 A.)—A. de W.

*Sulphonated resin and method of making same.* A. Horwitz. U.S.P. 1,373,886, 5.4.21. Appl., 19.11.18.

A SULPHONATED resin soluble in water is obtained by the treatment of a resin with sulphuric acid.—A. de W.

*Resins; Process for bleaching —.* G. Ruth and E. Asser. G.P. 329,186, 21.12.19.

THE resin is dissolved in a solvent, neutralised if necessary, and saturated, preferably warm, with hydrogen sulphide. Iron compounds present as impurities are thus precipitated.—J. H. L.

*Glycerides of aliphatic acids; Method for rendering soluble the solidified (polymerised) —.* O. Nagel. G.P. 331,870, 9.3.20.

SUBSEQUENT to or during the polymerisation process, colloids soluble in the desired solvent are added. Polymerised linseed oil after treatment with casein or vegetable gum will give with water or a hydro-

carbon solvent a solution or emulsion which can be applied in the same way as paint.—D. F. T.

*Resinous condensation products; Production of —.* Akt.-Ges. für Anilin-Fabr. G.P. (A) 332,334, 23.6.18, and (B) 332,391, 9.1.18.

(A) FORMALDEHYDE or substances capable of yielding formaldehyde are made to interact with halogen-substituted derivatives of naphthalene in the presence of acids. From  $\alpha$ -chloronaphthalene there are thus obtained clear elastic resins, soluble in benzene, chlorobenzene, fatty oils, or turpentine, and suitable for use in varnishes and for the preservation of leather. (B) Resinous products insoluble in alcohol, but soluble in benzene, turpentine, and linseed oil are obtained by the action of a metal halide on an additive compound of naphthalene with a halogen, or on halogen-substituted derivatives of hydro-naphthalenes either alone or mixed with aromatic hydrocarbons. Typical products are those obtained from the action of aluminium chloride on a solution of naphthalene tetrachloride in toluene, of ferric chloride on a mixture of 1-chloronaphthalene tetrachloride with naphthalene, and of antimony pentachloride on a solution of a similar mixture dissolved in tetrachloroethane.—D. F. T.

*Resin etc. from wood.* E.P. 160,482. See V.

*Lead oxides.* E.P. 137,288. See VII.

*Oil-proof concrete.* E.P. 145,673. See IX.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Amyrin and lupeol in the rubber from Ficus Vogelii.* A. J. Ultee. Ber., 1921, 54, 784—785.

SPENCE (J., 1907, 422) has described the isolation of  $\alpha$ - and  $\beta$ -albans, m.p. 201°—205° C. and 154° C. respectively, from *Ficus Vogelii*. The former is shown to be identical with  $\alpha$ -amyrin acetate, m.p. 218° C.; the latter contains lupeol, probably as the acetate. The terms,  $\alpha$ - and  $\beta$ -alban, should be deleted from the literature.—H. W.

#### PATENTS.

*Rubber goods and materials; Process for obtaining vulcanised — containing substances which are adversely affected by certain vulcanising processes.* S. J. Peachey and A. Skipsey. E.P. 160,499, 24.11.19.

FILLING materials, such as sawdust, cork dust, leather waste, and wool waste, which are adversely affected by ordinary vulcanisation, also lake pigments, can be used satisfactorily in compounding rubber which is to be vulcanised by the special method described in E.P. 129,826 (J., 1919, 688 A).—D. F. T.

*Rubber; Process of compounding lubricated sulphur and — and vulcanising.* D. Repony, Assr. to Manhattan Mfg. Co. U.S.P. 1,372,041, 22.3.21. Appl., 24.9.19.

SULPHUR, which has been treated with a lubricant, is introduced into the previously formed mixture of rubber and the other compounding ingredients.—D. F. T.

*Caoutchoucs; Pre-treatment of commercial artificial — which are difficult to work on the rolls owing to insufficient adhesiveness and elasticity.* Akkumulatoren-Fabr. A.-G. G.P. 329,171, 3.1.17.

THE material is exposed to a temperature above 100° C., and if necessary washed and kneaded at the same time. The product dried in the usual way exhibits when worked almost the same adhesiveness, elasticity, "nerve," and capacity for filling materials as natural rubber.—J. H. L.

*Rubber; Increasing the elasticity of vulcanisates from artificial or natural* —. Farbenfabr. vorm. F. Bayer und Co. G.P. 332,347, 16.1.19.

BEFORE vulcanisation, an aryl or aralkyl ether, e.g., 5% of dibenzyl or dixyl ether, is added to the rubber.—D. F. T.

## XV.—LEATHER; BONE; HORN; GLUE.

*Gallotannin.* XII. M. Nierenstein, C. W. Spiers, and A. Geake. Chem. Soc. Trans., 1921, 119, 275—286.

FURTHER evidence obtained on the constitution of gallotannin seems to throw doubt on the correctness of the pentadigalloylglucose formula of Fischer and Freudenberg (J., 1912, 503). The formation of ellagic acid from gallotannin occurs in two distinct stages, the first producing a maximum of 60%, and the second giving only traces of ellagic acid, but about 8% of dextrose. Similarly, partially hydrolysed gallotannin gave ellagic acid in two distinct stages, whereas from the pentadigalloylglucose formula simultaneous production of ellagic acid and dextrose would have been expected. Further, the methylated derivative of the above partially hydrolysed gallotannin, and also of gallotannin itself, gave on hydrolysis tetramethylglucose. These results are fundamentally opposed to the pentadigalloylglucose formula, since apparently four hydroxyl groups of the glucose in gallotannin are free unless the methylation with diazomethane had caused a replacement of the acyl groups, which, however, was shown to be excluded by the fact that dextrose, and not tetramethylglucose, was formed by the hydrolysis of the methylation product obtained by the action of diazomethane on a synthetic pentagalloylglucose.—G. F. M.

### PATENTS.

*Tanning material; Production of a* —. H. Hassler. G.P. 306,341, 9.12.13.

CRYSTALLINE sulphonic acids of unsubstituted tricyclic aromatic hydrocarbons, or the condensation products of aromatic sulphonic acids, may be used either alone, or mixed with other natural agents, for tanning hides. An acid reaction is maintained during the process, and the resulting leather is of a bright appearance. Suitable compounds are anthracenedisulphonic acids, and condensation products of naphthalenesulphonic acid, as well as sulphonic acids of phenanthrene and fluorene.—W. J. W.

*Patent leather; Process for hardening* —. J. J. Stöckly. G.P. 331,871, 14.4.18.

HARDENING of the surface of enamelled or patent leather is accelerated by treatment with ethyl alcohol. It is further advantageous to add an alkali to neutralise acidity developed during drying, which exerts a retarding action on the hardening.—W. J. W.

*Tanning material; Production of a* —. H. Renner and W. Moeller. G.P. 333,403, 12.12.18. Addn. to 262,333 (J., 1913, 878).

ACID resin or acid tar from the refining of mineral oils is treated with alkali or alkaline-earth sulphides, polysulphides, or hydrosulphides instead of with hydroxides or carbonates as described in the chief patent. If calcium (or barium) sulphide is employed, the precipitate of sulphate formed carries down with it the highly coloured impurities, and the clear liquor obtained after filtration can be used immediately for tanning, yielding a light-coloured leather.—L. A. C.

*Catgut; Sterilisation of* —. O. Frey. G.P. 331,169, 13.3.19.

THE intestinal skins are treated with solutions of diaminomethylacridinium chloride (Trypaflavin). They may further be dressed, e.g., with tannin or potassium iodide solution containing iodine.

—J. H. L.

*Catgut; Preparation of* —. C. Braun. G.P. 332,383, 1.8.18.

BY treatment of the intestines with a solution of chlorine or bromine, previous to or instead of the usual treatment with iodine solution, surface hardening is avoided and the disinfecting solution more easily penetrates into the body of the material.—B. V. S.

*Casein; Production of* — in a form suitable for the manufacture of translucent horn-like materials. R. Weiss. G.P. 331,440, 26.10.17.

AFTER separation of albumin from milk, the casein is precipitated with aluminium acetate, and then freed from fat by treatment with a mixture of alcohol and another solvent. Owing to the shrinkage caused by the aluminium acetate precipitation, enough moisture is retained by the casein to permit of its being moulded into a transparent, horny material.—W. J. W.

*Impregnating agents.* G.P. 302,531. See V.

*Cellulose compounds.* G.P. 332,203. See V.

*Dyeing furs.* G.P. 334,012. See VI.

*Waste water from tanneries.* G.P. 333,703. See XIX B.

## XVI.—SOILS; FERTILISERS.

*Soils; Adsorption phenomena in* —. VI. Determination of adsorbed bases, and their significance for processes which take place in the soil. D. J. Hissink. Versl. Landbouwk. Onderzoek. Rijkslandbouwproefstat., 1920, 144—150. Chem. Zentr., 1921, 92, 11., 594—595. (Cf. J., 1919, 918 A.)

BASES occur in the soil in two forms between which there is little interchange, viz., in an adsorbed form, probably as ions, easily replaced by other ions, e.g., by treatment with ammonium salts, and on the other hand in a form soluble in acids. The adsorbed bases form the supply immediately available for plant nutrition. To determine adsorbed lime or magnesia, 25 g. of soil (or 50 g. in the case of humic sandy soil) is treated with 100 c.c. of warm N/1 sodium chloride solution and left to stand. On the following day it is filtered and washed with N/1 sodium chloride solution until two litre flasks have been filled in turn with the filtrate and washings. The difference between the lime-content of the first and second litres represents the amount of adsorbed lime in the soil, and similarly for magnesia. To determine adsorbed soda and potash 25 g. of soil is treated as above with N/1 ammonium chloride solution, and two successive lots of  $\frac{1}{2}$  l. each of filtrate are collected, the difference between their contents of potash or soda representing the amount adsorbed in the soil. Average results for a number of clay soils showed 30 mg.-equivalents of calcium ions, 5 of magnesium, 0.8 of potassium, and 2.5 of sodium ions per 100 g. of soil; i.e., of 100 adsorbed cations 79 were calcium, 13 magnesium, 2 potassium, and 6 sodium ions. Importance is attached to the "state of saturation" of a soil, i.e., the ratio of the number of adsorbed cations to the number which the soil is capable of adsorbing, but there is at present no accurate method of determining the latter value. In moist sea climates the soils are

unsaturated and are liable to become "acid." A soil which had been spoiled by flooding with sea water contained per 100 adsorbed cations 56.9 of calcium, 20.2 of magnesium, 3.7 of potassium, and 19.2 of sodium. The flooding had also spoiled the organic matter, by converting calcium humate gels into sodium humate soils.—J. H. L.

*Soil; Experiments on chemical and bacteriological changes in —.* F. Münter. *Landw. Jahrb.*, 1920, 55, 62—138. *Chem. Zentr.*, 1921, 92, II., 68—69.

MANY experiments were performed upon the influence of saline fertilisers on soil. Monobasic calcium phosphate raised the solubility of silica and lessened the solubility of iron and calcium salts. Neutral and basic calcium salts lessened the solubility of silica. The solubility of tribasic calcium phosphate in ammonium chloride was increased in soils poor in lime and rich in silica, whereas the solubility of calcium carbonate in ammonium chloride was lessened. The solubility of lime in water was increased by the presence of potassium chloride, potassium sulphate, and ammonium sulphate. The solubility of magnesia in water and in dilute hydrochloric acid was increased by acid calcium phosphate. In soils containing much iron and clay, the addition of caustic lime diminished the solubility of phosphoric acid in dilute hydrochloric acid. In aqueous solution phosphoric acid is absorbed by lime, iron, and clay, and then behaves similarly to tribasic calcium phosphate. Whilst almost all added phosphoric acid can be recovered by acid from soils rich in lime, 70% of it is retained by soils poor in lime but rich in iron and clay. With regard to the solubility of phosphates, it was found that although ammonium oxalate and acetate could dissolve many of them, yet the best and quickest solvent was citric acid. The influence of moisture upon the nitrogenous changes was investigated and it was found that the heavier the soil, the higher must its water content be for the same amount of decomposition to take place in the organic nitrogenous substances. The rapidity of the formation of ammonia and nitric acid in presence of little moisture increased as the lightness of the soil increased. In soils with much moisture the highest nitrate content was found in sandy soils, because in heavy soils nitrate was decomposed, as much as 32% being lost in a loam soil compared with 9% in a sandy soil. The presence of zeolites caused a sandy soil to behave like a loam soil with respect to ammonification and nitrification. Actinomycetes in soil were found to obtain their nitrogen supply from inorganic as well as organic sources and their carbon supply from carbohydrates and organic acids. Urea, thiourea, and dicyanodiamide served only as sources of nitrogen. Alkali salts were tolerated up to 5%; larger amounts of the alkaline-earths were rendered harmless owing to conversion into carbonates. These fungi effected ammonification, but nitrification was only slight, and evolution of nitrogen did not occur at all; neither did nitrogen fixation, even in the presence of other organisms.—J. H. J.

*Soils; Relation of the calcium content of some Kansas — to the soil reaction as determined by the electrometric titration.* C. O. Swanson, W. L. Latshaw, and E. L. Tague. *J. Agric. Res.*, 1921, 20, 855—868.

THE total calcium and calcium in forms soluble in  $N/5$  and  $N/1$  hydrochloric acid, present in a number of Kansas soils, and the hydrogen-ion concentration of suspensions of 10 g. of the soil in 100 c.c. of water were determined. In soils with a high calcium content, a larger percentage of the calcium was in forms soluble in the dilute hydrochloric acid than in soils with a low calcium con-

tent. As a rule soils with a high calcium content had a higher hydroxyl-ion concentration than those with a low calcium content, but the amount of  $N/25$  calcium hydroxide necessary to change a soil from a lower to a higher hydroxyl-ion concentration was dependent rather upon the amount of colloidal clay present than upon the calcium content. The amount of  $N/25$  calcium hydroxide required to change the  $pH$  value of acid soils to 7, when calculated in terms of calcium carbonate per acre, compared moderately well with the results from the Hopkins method for determining lime requirements. Subsoils, as a rule, had a higher calcium content than surface soils and required more calcium hydroxide to change their  $pH$  values over a given range.—W. G.

*Azotobacter.* Kayser. *Comptes rend.*, 1921, 172, 939—940. (*Cf. J.*, 1921, 155 A.)

A FURTHER study of the effect of colour on Azotobacter at different stages of its culture and the influence of temperature in varying the results. At 27° C. the generations from yellow and blue growths gave practically the same results in respect to total nitrogen fixed and nitrogen fixed per gram of mannitol consumed. The utilisation of mannitol became less satisfactory as the time of incubation was extended. It was more satisfactory at 8°—16° C. than at 27° C., although the total nitrogen fixed at the lower temperature was much less.

—W. G.

*Arsenic, antimony, and fluorine compounds; Poisonous action of — on plant cultures.* A. Wöber. *Angew. Botanik*, 1920, 2, 161—178. *Chem. Zentr.*, 1921, 92, I., 35—36.

AMONG the plants most susceptible to poisons were beans, peas, barley, oats, wheat, maize, and rye. In water cultures 0.01 g. of arsenious oxide per litre had a very poisonous effect, arsenic pentoxide being less poisonous, and at a concentration of 0.1 g. per l. all plants died. Antimony compounds had much lower toxic powers than this. Sodium fluoride had an injurious effect at a strength of 0.1 g. per l. When dusted on soil arsenious oxide had a less poisonous effect than arsenic pentoxide, and antimonious oxide was still less poisonous and was about on a par with sodium fluoride. When sprinkled on green plants, solutions of arsenic acid were more injurious than those of arsenious acid, whereas their sodium salts behaved in a converse manner. Sodium fluoride had a caustic effect in 1% strength.—J. H. J.

#### PATENTS.

*Phosphatic fertiliser; Process for the manufacture of —.* F. J. Tromp. U.S.P. 1,372,051, 22.3.21. Appl., 11.6.19.

FOR the production of phosphates soluble in citric acid, iron phosphate or aluminium phosphate is treated with a mixture of lime and a caustifiable compound of an alkali metal in the presence of water.—D. F. T.

*Fertilisers; Manufacture of —.* Badische Anilin- und Soda-Fabr. G.P. 333,671, 31.3.18. Addn. to 332,114 (J., 1921, 315 A.)

COARSELY crushed superphosphate is mixed directly with ammonium bicarbonate and the mixture ground.—A. R. P.

*Seeds; Treatment of —.* Chem. Fabr. L. Meyer. G.P. 327,310, 10.8.19. Addn. to 321,797 (J., 1920, 667 A.)

PHENOLS may be used in admixture with mercury or silver salts without injuring the germinating capacity of the seeds.—J. H. J.



## XVII.—SUGARS; STARCHES; GUMS.

*Clerget method* [of determining sucrose by double polarisation]; Application of the — to dilute solutions. R. F. Jackson and C. L. Gillis. *Int. Sugar J.*, 1921, 23, 217—220.

REPLYING to Browne's criticism (J., 1921, 271 A), it is pointed out that, while the optical rotation of sucrose in the presence of ammonium chloride, sodium chloride, etc. (J., 1920, 634 A) is increased in dilute solution, that of invert sugar, on the other hand, is diminished, the effect of the salt added being compensating (within the limit of error) in the two polarisations. In any case, the effect is small, a value, for example, of  $19.17^\circ$  S. being obtained in the case of 5 g. of sucrose in the presence of 3.392 g. of ammonium chloride per 100 c.c., whereas the value would have been  $19.12^\circ$  if the rotation had been strictly proportional to the concentration. Results of analyses are reproduced showing the average error in the proposed method of neutral polarisation to be only 0.02% in the case of pure solutions of sucrose containing 3—7 g. per 100 c.c.—J. P. O.

*Sugars; Iodometric determination of —.* J. L. Baker and H. F. E. Hulton. *Biochem. J.*, 1920, 14, 754—756.

THE method of Willstätter and Schüdel (J., 1918, 556 A) is accurate. The reagents must be added in the order—sugar, iodine, alkali, and the time of oxidation need be only 3—5 mins. (Cf. J.C.S., May.)—J. C. D.

*Sugar analysis; Iodometric determination of copper and its use in —.* I. Equilibria in the reaction between copper sulphate and potassium iodide. P. A. Shaffer and A. F. Hartmann. *J. Biol. Chem.*, 1921, 45, 349—364.

THE influence of the equilibrium between copper sulphate and potassium iodide on the estimation of sugars has been investigated. For the determination of cupric salts potassium iodide must be added to give a final concentration of about 0.25 M (4—5 g. per 100 c.c.). For the determination of cuprous salts the final concentration of copper and of iodide should not exceed about 5 millimolar each. A number of the methods in use (Gooch and Heath, J., 1907, 991; Maclean, J. *Physiol.*, 1916, 1, 168; *Biochem. J.*, 1919, 13, 135; Scales, J., 1915, 1264) comply with the requirements. (Cf. J.C.S., May.)—J. C. D.

*Sugar analysis; Iodometric determination of copper and its use in —.* II. Methods for the determination of reducing sugars in blood, urine, milk, and other solutions. P. A. Shaffer and A. F. Hartmann. *J. Biol. Chem.*, 1921, 45, 365—390.

THE principles outlined in a previous paper (cf. *supra*) have been applied to the methods for the estimation of reducing sugars in biological fluids.—J. C. D.

*Polysaccharides. II. Constitution of diamylose.* P. Karrer and C. Nägeli. *Helv. Chim. Acta*, 1921, 4, 169—173. (Cf. J., 1920, 795 A.)

ACETYL bromide at the ordinary temperature converts  $\alpha$ -tetra-amylose (cf. Pringsheim and Langhans, J., 1912, 1001) first into acetylated  $\alpha$ -diamylose and then into acetobromomaltose, which is obtained in quantitative yield.  $\alpha$ -Diamylose is therefore an anhydride of maltose and is the first known anhydride of a disaccharide. (Cf. J.C.S., May.)—T. H. P.

*Polysaccharides. IV. Degradation of potato starch.* P. Karrer and C. Nägeli. *Helv. Chim. Acta*, 1921, 4, 185—202.

THE experiments of Karrer (J., 1920, 795 A) have

been completed and extended. Methylation of purified potato starch by means of methyl sulphate and baryta water leads to a methylostarch having the molecular weight in water 990—1026, or, allowing for inorganic matter present, 1200 at most. Aqueous or chloroform solutions of this compound contain colloidal particles, but are rendered optically empty by ultra-filtration; these are, therefore, true crystalline solutions, and they may be evaporated to dryness in a vacuum without alteration in the properties of the solute. This methylostarch,  $C_6H_7O_2(OCH_3)_2$ , is attacked neither by diastase nor by ptyalin; *Bacillus macerans*, which degrades ordinary starch to crystalline dextrins, does not develop in its solutions or cause its depolymerisation. The authors consider that the starch molecule contains not more than six dextrose residues united by chemically normal linkings; the number may, indeed, be less than six, since it is not certain that the limits of depolymerisation are reached by the methylation. (Cf. J.C.S., May.)—T. H. P.

*Polysaccharides. VI. Constitution of starch and glycogen.* P. Karrer and C. Nägeli. *Helv. Chim. Acta*, 1921, 4, 263—269.

Dry potato starch is attacked by acetyl bromide at the ordinary temperature, with formation of acetobromomaltose in large proportions. As experiments with other polysaccharides have shown that the glucosidic linking is stable towards acetyl bromide, it is assumed that in the starch molecule not more than two dextrose residues are united by normal glucosidic valencies. Starch may also be hydrolysed, with formation of well-defined products, by enzymes, by acid, and by *Bacillus macerans*, and consideration of these reactions indicates that starch is a polymerised maltose anhydride (diamylose). The degree of polymerisation is probably not very high, the high value usually attributed to the molecular weight of starch being presumed, erroneously, from its insolubility. Glycogen also must be regarded as a polymerised diamylose, the degree of polymerisation being different from that of starch.—T. H. P.

"Kelpchar." Turrentine and others. See IIb.

*Polysaccharides.* Karrer and Widmer. See V.

*Soluble carbohydrates in pinewood.* Schmidt-Nielsen. See V.

*Fats from carbohydrates.* Witzemann. See XII.

## PATENTS.

[Sugar juices and other] liquids or liquors; Process for the purification and separation or filtration of —. Filtering, decolorising, and purifying processes for liquids and liquors, juices, liquefied bodies, and the like. J. N. A. Sauer. E.P. (A) 155,609 and (s) 155,611, 20.6.19.

(A) IN a systematic process successive portions of the liquid, at progressive stages of purification, are passed in turn through a filter containing highly active, finely divided decolorising carbon. The filter is then recharged with fresh carbon (which may first be mixed with the most advanced liquid portion and transferred to the filter with the latter), and the cycle of operations is repeated, each portion of liquid being advanced one stage at every cycle. Any portion may be passed several times through the filter if necessary. The raw liquid introduced as the last portion in each cycle may have received a preliminary purification, and instead of being merely passed through the filter containing the nearly exhausted carbon it may be mixed with the latter and filtered. (s) IN the treatment of liquids with highly active, finely divided decolorising carbon, instead of employing just the amount necessary for one quantity of liquid so that the



carbon is spent thereby, a considerably greater amount is used so that it will serve for several successive quantities of liquid of the same degree of purity or colour. The time required for the treatment of a given quantity of liquid is very much reduced by this means.—J. H. L.

*Sugar; Decolorising and purifying* —. D. Grant. E.P. 159,640, 6.12.19.

Raw or incompletely refined sugar is mixed with a suitable proportion (0.5–6%) of dry active decolorising carbon, or kieselguhr, or both. In this form the sugar may be shipped in bulk and transported pneumatically, and it undergoes relatively little bacterial deterioration during storage. The refining of sugar thus treated is more economical in respect of purifying materials and time than in the case of untreated raw sugar.—J. H. L.

*Sugar-cane and similar materials; Treatment of* —. F. H. Lindenberg, Assr. to The Jeffrey Manufacturing Co. U.S.P. 1,369,180, 22.2.21. Appl., 13.1.16.

For the complete extraction of the juice the cane is thoroughly disintegrated so as to form a homogeneous pulp from which the fibrous matter is afterwards separated. The disintegration of the cane may be carried out in stages.—J. H. L.

*Sugar-beets; Process of making a palatable syrup from* —. S. F. Sherwood. U.S.P. 1,370,372, 1.3.21. Appl., 19.5.20.

SUGAR-BEETS are heated in water and the extract, separated from the solid matter, is heated at about 100° C. in an autoclave for 1 hr., in the course of which considerable quantities of steam are blown off at intervals of about 15 mins., whereby objectionable flavouring substances are eliminated.

—J. H. L.

*Sugar; Manufacture of — direct from the juice.* E. Delafond. U.S.P. 1,371,997, 15.3.21. Appl., 24.7.20.

SUGAR juices and syrups are purified by passing them between electrodes, and at the same time keeping them in sufficiently rapid motion to prevent any substantial deposition on the electrodes.

—J. H. L.

*Sugar; Counter-current apparatus, in particular for the extraction of plant slices containing* —. Maschinen und Werkzeugfabr. A.-G. vorm. A. Paschen. G.P. 331,129, 17.12.19.

IN apparatus with separating chambers for the temporary separation of the liquid from the slices by means of overflow walls, according to G.P. 267,133, 269,925, and 278,067 (cf. U.S.P. 1,077,293 and 1,134,152; J., 1913, 1123; 1915, 567), the separating chambers are provided with a wide outlet orifice for the liquid. There is no danger of slices entering through this orifice, owing to the strong counter-current of liquid.—J. H. L.

*Sugar for consumption; Production of — from mossecuites and their drainings.* A. Müller. G.P. 329,890, 17.1.20.

THE boiling of the massecuite is arrested at a higher moisture content than usual, and it is brought to the density necessary for satisfactory centrifuging by adding, either in the vacuum pan or preferably in the cooler, dry or moist sugar, massecuite of low moisture content, or a mixture of grainy sugar and thick syrup or the latter alone. The strongly coloured solutions obtained in boiling out or steaming out the evaporating plant are worked up separately. With careful working, sugars for direct consumption may be obtained even from second products without re-melting.

—J. H. L.

*Molasses; Process for the desaccharification of* —. A. Gräntzdörffer. G.P. 330,640, 11.4.20. Addn. to 311,212 (J., 1919, 550 A).

THE addition of second syrup and dissolved sugar to first products in the vacuum pan may be made before the formation of grain in the latter. The whole of the centrifugal runnings from first product sugar may be added to the next charge of first syrup, the major part being added before the formation of grain and the rest at the close of the boiling process.—J. H. L.

*Root fruits [beetroots]; Method for extracting juices from* —. O. Mengelbier, Assr. to the Chemical Foundation, Inc. U.S.P. 1,372,891, 29.3.21. Appl., 11.5.14.

SEE F.P. 470,980 of 1914; J., 1915, 192.

*Sugar beets.* G. P. 324,641 and 325,392. See XIX A.

## XVIII.—FERMENTATION INDUSTRIES.

*Barley; Harvesting, storage and drying of* —. A. Cluss, W. Kluger, and V. Koudelka. Z. ges. Brauw., 1921, 4–18.

EXPERIMENTS similar to those of 1913 (J., 1921, 22 A) were carried out in 1914 with the same kind of barley. Owing to difference in weather and other conditions the 1914 barley was drier and richer in starch and extract than the 1913 crop, and its germinative power developed much more rapidly. By deferring the harvesting of the grain until it was "dead ripe" (a week after it had attained "full ripeness") the brewing quality of the barley was found to be improved. The advantages of drying the grain artificially, although well marked, were much less so than in the case of the previous crop, and were practically the same whether the drying was carried out immediately after threshing or after 6 weeks' storage. Storage in the ear proved less advantageous than in the earlier experiments.

—J. H. L.

*Hops; Drying of* —. A. H. Burgess. J. Inst. Brew., 1921, 27, 180–184.

TO obtain data respecting the influence of various factors on the drying of hops under practical conditions, 11 kilns of various types were visited and records were made of 15 oastings. The data thus obtained are tabulated. The depth of the hops on the kilns varied from 1½ to 19 ins., artificial draught being used in most cases for the deeper loadings. The average temperature throughout the process ranged from 57° to 84° C., the most usual being about 70° C. The highest maximum recorded was 103° C. and the product in this case was considered satisfactory. The time required to complete the drying varied from 8½ to 15½ hrs., average 10½ hrs. The time taken does not depend solely on temperature, depth of load, and moisture-content of the hops. Hops wet with rain dried in some cases as rapidly as those picked in fine weather, and some oastings at comparatively high temperatures dried more slowly than others worked at a lower temperature. The most important factors are probably temperature and air supply, but the latter could not be estimated with any approach to accuracy.

—J. H. L.

*Yeasts; Break of* —. H. Lüers and R. Heuss. Z. ges. Brauw., 1921, 18–22.

THE flocculation or "break" of yeast, in aqueous suspensions, is closely related to the viscosity of the suspensions. Calcium or cerium chloride increased the viscosity and promoted flocculation to a much greater extent than equimolecular proportions of potassium chloride. The viscosity of 12%

suspensions of washed living yeast varied greatly with the reaction of the liquid, attaining a very well-defined maximum point when the reaction was slightly acid ( $p_H$  = about 4), and this point corresponded also with the maximum of flocculation. Similar suspensions of yeast killed by chloroform or heat showed no flocculation, and their viscosities were relatively low and varied to only a slight extent with the reaction of the liquid.—J. H. L.

*Yeast; Cultivation of — in solutions of purified nutrients.* M. B. MacDonald and E. V. McCollum. *J. Biol. Chem.*, 1921, 45, 307—311.

It appears probable that yeast can synthesise the growth-stimulating principle known as vitamin B for purposes of its own growth.—J. C. D.

*Iron; Determination of — in wines.* L. Mathieu. *Ann. Chim. Analyt.*, 1921, 3, 106. (*Cf. Malvezin and Rivalland, J.*, 1921, 316 A.)

The author prefers the colorimetric method, using potassium thiocyanate; the ash of the wine is dissolved in warm 10% sulphuric acid, any ferrous iron oxidised by the addition of a few drops of nitric acid, and the solution then treated with thiocyanate.—W. P. S.

*Sulphite spirit; Acetaldehyde content of —.* R. Sieber. *Chem.-Zeit.*, 1921, 45, 349—350.

The quantity of aldehyde present in sulphite spirit is proportional to the amount of sulphurous acid in the wort before fermentation; with an  $SO_2$  content rising from 0.132 to 0.330 g. per 100 c.c., the aldehyde increases from 0.044 to 0.220 g. per litre of spirit. The  $SO_2$  content does not alter appreciably during fermentation, and the portion which is presumably combined with the aldehyde is resistant to oxidation by aeration.—W. P. S.

*Aerobacter.* Kayser. See XVI.

*Toxicity of phenol towards yeast.* (1) Fraser. (2) Fulmer. See XIXs.

#### PATENTS.

*Barley; Process for removing testinic acids and their compounds from —.* Nathan-Institut A.-G. G.P. 330,341, 24.5.17. Addn. to 327,498 (J., 1921, 235 A.)

BARLEY is treated in the steep with dilute alkalis to dissolve the testinic acid, and then completely freed from the latter by washing. The treatment improves the flavour, head-retaining power, and stability of beer produced from the barley, and also renders low-grade barleys suitable for brewing. It may be applied also to oats.—J. H. L.

*Glycerin; Production of — by fermentation.* A. Koch. G.P. 331,694, 20.11.17.

SUGAR solutions of high concentration, e.g., 40%, are fermented by large quantities of yeast, and alcohol is removed either continuously by means of a vacuum or from time to time by distillation under reduced pressure. The yield of glycerin amounts to at least 15% of the sugar fermented.—J. H. L.

*Adhesive for belts and the like [from fermentation residues].* H. Haselberger. G.P. 328,881, 31.7.18.

RESIDUES from the distillation of fermented liquids, containing glycerol and other hygroscopic substances, are treated with a protective agent against rust and made into a syrup with water or other liquid.—J. H. L.

*Alcohol distillation.* U.S.P. 1,372,465. See IIa.

*Sugar beets.* G.P. 324,641 and 325,392. See XIXa.

#### XIXa.—FOODS.

*Milk; Vitamin content of —.* F. G. Hopkins. *Biochem. J.*, 1920, 14, 721—724.

The author has repeated and confirmed the results of his earlier experiments on the influence of the vitamins in very small quantities of milk on the growth of rats (*cf. J. Physiol.*, 1912, 44, 425).

—J. C. D.

*Vitamin A in butter; Effects of heat and aeration upon —.* F. G. Hopkins. *Biochem. J.*, 1920, 14, 725—733.

The refining processes ordinarily employed for ground-nut (arachis) oil and palm kernel oil do not appear to affect the amount of vitamin present, which is very small. The vitamin present in butter fat is not appreciably destroyed by heat alone at temperatures up to 120° C., but is readily rendered inactive by aeration of the hot fat. The vitamin would appear to be a substance prone to oxidative change.—J. C. D.

*Butter; Effect of heat and oxygen on the nutritive value of —.* J. C. Drummond and K. H. Coward. *Biochem. J.*, 1920, 14, 734—739.

The vitamin in butter is rapidly destroyed by contact with air at high temperatures. Destruction occurs at temperatures as low as 37° C. if contact with air or oxygen is extensive. (*Cf. supra.*) —J. C. D.

*Vitamin A in fats; Action of ozone on —.* S. S. Zilva. *Biochem. J.*, 1920, 14, 740—741.

The vitamin A in butter and cod liver oil is rapidly destroyed by exposure to ozone. (*Cf. supra.*) —J. C. D.

*Lard; Nutritive value of —.* J. C. Drummond, J. Golding, S. S. Zilva, and K. H. Coward. *Biochem. J.*, 1920, 14, 742—753. (*Cf. J.*, 1921, 81 T.)

The pig resembles other animals which can store up the vitamin A in their body fats, but this is only effected when the diet contains ample amounts of that factor. Fat derived from grass-fed pigs is fairly rich in the vitamin, but the amount may be considerably reduced by the processes of lard manufacture, which frequently involve contact of the hot fat with air.—J. C. D.

*Vitamin A; Differentiation of — from yellow plant pigments.* M. Stephenson. *Biochem. J.*, 1920, 14, 715—720.

The author confirms Drummond's observation (J., 1919, 435 A) that the vitamin A is not one of the lipochromes. Butter fat filtered through charcoal loses its pigment, but retains the vitamin.—J. C. D.

*Diets free from vitamin A; Critique of experiments with —.* T. B. Osborne and L. B. Mendel. *J. Biol. Chem.*, 1921, 45, 277—288.

A GENERAL discussion of the technique employed for testing substances for the presence of the vitamin A, with especial reference to the divergent results obtained by different workers.—J. C. D.

*Hay; Results of investigation into the self-heating of —.* G. Laupper. *Landw. Jahrb. Schweiz*, 1920, 34, 1—54. *Chem. Zentr.*, 1921, 92, I., 162—163.

The view put forward is that the moisture from withered hay in a stack is able to activate the oxidases present in dried green hay with consequent chemical action and heat evolution. Micro-organisms play no part in the process. The following sequence of events was ascertained: At

20°—35°, activation of the oxidases, commencement of heating; at 35°—45°, caramelisation of sugar; at 45°—70°, evolution of ammonia and formic acid; at 60°—70°, acceleration of the exothermic reaction; at 70°—90°, decomposition of pectins, pungent odour; at 90°—100°, first decomposition of protein, formation of hydrogen sulphide and furfural; at 110°—170°, formation of nitric acid by oxidation of ammonia, explosive union between ammonium nitrate and caramel carbon; at 170°—250°, rapid progress of the exothermic reaction; at 250°—280°, decomposition of cellulose and protein, formation of hydrogen sulphide and furfural, ignition of pyrophoric iron; at 300°, region of inflammability through access of oxygen; at 320°—340° C., final decomposition of carbohydrates, formation of furfural and pyrophoric manganese. The injection of carbon dioxide into the stack does not remove the danger of fire.—J. H. J.

*Reducing sugars in milk etc.* Shaffer and Hartmann. See XVII.

*Lactic acid in baking powder.* Hartwig and Saar. See XX.

#### PATENTS.

*Concentration of fruit juices in the manufacture of grape honey and fruit syrups; Process and apparatus for the —.* E. Barbet et Fils et Cie. E.P. 135,175, 6.11.19. Conv., 9.11.18.

For the economical concentration of fruit juices in vacuum multiple-effect apparatus, the different effects are used for different juices according to their sensitiveness to heat. Thus the first effect may be used for sulphured juice from white grapes which, for the end in view, is not injured by boiling at 70° C. or above, whilst the second effect is used for the more sensitive juice from red grapes, which would be impaired by boiling at temperatures above 55° C. Each effect may comprise more than one evaporator, and the concentration of each juice may be carried out in two stages between which the juice is allowed to cool and deposit tartar. An arrangement of apparatus for carrying out the process is described.—J. H. L.

*Fruit and other vegetable matter; Method of and apparatus for the extraction of juices, pectin, and the like from —.* J. Nicholson. E.P. 159,311, 24.11.19.

POMACE or other suitable material, preferably freed from sugar, is introduced into a strainer immersed in water, and simultaneously heated and agitated, e.g., by injection of steam, to obtain an extract suitable for use in the manufacture of jam. The apparatus comprises a bag-shaped strainer of copper gauze, suspended in a pulp container, which is provided with a steam-tight cover and may be fitted with perforated steam pipes around and within the strainer.—J. H. L.

*Milk food.* C. S. Townsend. E.P. 159,362, 10.12.19.

LIQUEFIED jelly obtained from *Chondrus crispus* (Irish moss) is emulsified with whole-cream milk, and the product is dried.—J. H. L.

*Food and food products; Preparation of —.* J. G. Goodhue and H. L. Trube. U.S.P. (A) 1,365,909 and (B) 1,365,910, 18.1.21. Appl., 4.10. and 19.11.17.

(A) A DRYING chamber is provided with passages for conveying a number of air currents over material to be dried and back again. Means are provided for inducing the air currents and for heating them independently of the drying chamber. Mechanical means are provided within the chamber for keeping the foodstuff moving forward during the process. (B) A drying chamber is divided into smaller chambers by transverse partitions. Each chamber contains a receptacle for the material to be dried with

its end placed at the transverse partition, which is provided with openings placed alternately on opposite sides of the receptacles and forming passages between adjacent chambers, so as to provide a path for a drying current across the receptacles. An air current is circulated by means of a blower at one end of the main chamber and a heater at the opposite end.—J. H. J.

*Flour and other cereal products; Process of bleaching and maturing —.* J. C. Baker. U.S.P. 1,367,530, 8.2.21. Appl., 14.2.20.

NITROGEN trichloride gas is passed through the flour.—J. H. J.

*Coffee extract; Method of making water-soluble — and the product. Method of adding segregated aromas to coffee extract.* D. S. Pratt and C. W. Trigg, Assrs. to J. E. King. U.S.P. (A) 1,367,715 and (B) 1,367,716, 8.2.21. Appl., 31.5.18.

(A) THE aroma is separated from a coffee infusion and is brought into contact with lactose which absorbs it. The infusion is evaporated to dryness and the impregnated lactose is mixed with the residue. (B) A coffee extract is sprayed with a solution of coffee aroma in a volatile solvent.

—J. H. J.

*Aromatic and flavouring constituents; Process for recovery of —. Process for recovery of escaping aromas of food etc. Recovery of aromas etc. escaping during comminution of coffee-beans.* C. W. Trigg, Assr. to J. E. King. U.S.P. (A) 1,367,721, (B) 1,367,725, and (C) 1,367,726, 8.2.21. Appl., (A, B) 19.11.17, (C) 31.5.18.

(A) AN infusion of roasted ground coffee is evaporated to dryness, the vapour given off being condensed. From the condensed product the cafeeol is separated by a solvent and is added to the coffee extract, the solvent being subsequently removed. (B) A coffee infusion is evaporated in a vacuum, the vapours condensed, and the cafeeol extracted. The chamber is then exhausted and the gases passed into a closed chamber where they are compressed above atmospheric pressure and passed through a cafeeol solution, and then passed into a coffee extract chamber under pressure. When the extract has absorbed the cafeeol solution, the pressure is reduced and the solvent allowed to escape. (C) Roasted coffee beans are ground in a closed chamber from which air is excluded. An inert gas is passed through the chamber to remove the aroma and brought into contact with a water-soluble coffee extract, which absorbs the aroma.—J. H. J.

*Sterilizing milk and other liquids; Process and apparatus for —.* O. Lobeck, Assr. to The Chemical Foundation. U.S.P. 1,369,345, 22.2.21. Appl., 18.2.15.

THE milk, by a rotary device, is formed into an extremely thin layer, and in this condition is heated under reduced pressure at a temperature below its boiling point. (Cf. G.P. 306,924; J., 1918, 669 A.)—J. H. L.

*Drying food products and other materials; Apparatus for dehydrating or —.* W. D. Edwards. U.S.P. 1,369,411, 22.2.21. Appl., 26.2.19.

THE apparatus comprises a number of independent chambers arranged side by side, perforated supports for the material to be dried extending diagonally across the chambers so as to divide each into two compartments larger at one end than the other, and means for introducing air into all the chambers at one end, for controlling the air-inlet of each chamber, and for introducing moisture into the air at the inlet ends of the chambers.

—J. H. L.

*Fish; Food obtained from — and process of making the same.* S. Satow. U.S.P. 1,370,049, 1.3.21. Appl., 5.5.17.

THE cooked residue from the manufacture of fish oil is crushed or ground to a paste, dried in *vacuo* below 50° F. (10° C.), ground to powder, and subjected to the action of a proteolytic enzyme, after which the soluble protein is extracted with water, evaporated in *vacuo* at a low temperature, and powdered.—J. H. L.

*Evaporated vegetables; Method of preparing —.* R. S. Wittenberg, Assr. to The Pacific Evaporator Co. U.S.P. 1,372,112, 22.3.21. Appl., 17.4.19.

THE vegetables are sliced, the cut surfaces subjected to the abrasive action of a jet of fluid of such force and so directed as to remove the free substances exposed by the slicing, and the slices then dehydrated.—A. de W.

*Culinary product. Edible oil material and process of producing same.* C. Ellis. U.S.P. (A) 1,372,614 and (B) 1,372,615, 22.3.21. Appl., 25.1.19 and 24.4.18.

(A) A food product (nut butter) of a consistency about that of butter is produced by mixing several oils (coconut and peanut oils) together and completely hydrogenating them with the aid of a catalyst. (B) A product of lard- or butter-like consistency is formed from a mixture of a normally liquid nut oil, hydrogenated to a hardness at least as great as that of tallow, with a substantially greater amount of an unhydrogenated nut oil having an iodine value below 25 and a saponification value above 240.—B. M. V.

*Sugar beets; Process for purifying and deodorising comminuted —.* Betavit-Ges. m.b.H. G.P. (A) 324,641, 14.12.17, and (B) 325,392, 12.11.18.

(A) STEAM at 100°–104° C. is passed through the material until the flavour and odour of beets have been removed. The product has a pleasant vanilla-like odour and may be used for the production of beer or other beverages. Sugar beet flour may be obtained by atomising and desiccating the juice, and the slices, separated from the juice, may be used for the preparation of foodstuffs or fodder. (B) A current of dry gas, e.g., air or carbon dioxide, is employed instead of steam, whereby dilution of the material is avoided.—J. H. L.

*Maize meal or maize grits; Removal of bitter substances from —.* P. Schuffelhauer and O. Fauser. G.P. 328,425, 1.11.16.

THE material, enclosed in a bag or similar container, is treated several times with boiling brine and then allowed to cool and taken out of the container. The skin which has formed on the surface is removed and the material ground and dried in hot air. The maize oil may be extracted from the brine and the skin.—J. H. L.

*Fodder grasses; Process for rendering withered — stable by spontaneous fermentation.* "Herba" A.-G. G.P. 328,781, 31.5.17. (Cf. G.P. 305,562; J., 1918, 960 A.)

IN order to secure uniform fermentation throughout the whole mass of fodder, the layers of which are exposed to the cooling influence of the floor, walls, and roof of the containing chamber, the fodder is treated with lactic bacteria; usually 150–180 c.c. of a pure culture in whey is sufficient for 1 cb. m. of fodder.—J. H. L.

*Straw; Process for the preparation of digested —, in which the production of waste liquors and losses by washing are avoided.* W. Thomann. G.P. 328,782, 16.5.18.

THE alkaline digested straw is neutralised with son-

wbey, sour buttermilk, or sour skim milk. The lactates formed improve the flavour of the fodder.—J. H. L.

*Straw (e.g., from cereals or leguminous plants); Production of fodder from — by digestion with ammonia.* E. Beckmann. G.P. 332,363, 25.2.19.

THE comminuted straw is digested below 80° C. with aqueous ammonia, preferably of 0.5–8% concentration, or with equivalent quantities of ammonium salts or mixtures which produce ammonia.—J. H. L.

*Fodder from straw; Preparation of a —.* Veredelungsges. für Nahrungs- und Futtermittel m.b.H. G.P. 333,746, 29.3.18. Addn. to 305,641 (J., 1919, 789 A.).

THE straw is digested with a solution of an alkali sulphide, with or without the addition of an alkali hydroxide instead of with an alkali solution as described in the chief patent.—L. A. C.

*Fruit and vegetable refuse of all kinds; Complete utilisation of —.* Malz- und Nahrungsmittelwerke A.-G. G.P. 331,005, 9.6.18.

WASTE materials such as pea shells, banana skins, and the like, are crushed and then extracted by boiling and pressure if necessary. The extract is concentrated and employed as a foodstuff and the residue is further digested with alkalis in order to obtain a liquid cattle fodder and a fibrous residue. Fibres thus obtained from asparagus waste can be used for textile purposes.—J. H. L.

*Quinoa; Preparation of a food product rich in protein from —.* E. Heilmann. G.P. 331,547, 11.8.18.

A FOODSTUFF and also an oil and saponin are obtained by drying the finely ground material below 60° C., extracting with dry ether or other fat solvent, and then treating the residue with alcohol, e.g., methyl alcohol, at 60° C. or under to extract the saponin.—J. H. L.

*Bread; Process for increasing the yield of —.* E. C. Sutherland, Assr. to Naaml. Vennoots. Ind. Maatschappij v.h. Noury en van der Lande. U.S.P. 1,372,842, 29.3.21. Appl., 18.9.17.

SEE E.P. 115,410 of 1917; J., 1919, 266 A.

## XIXs.—WATER PURIFICATION; SANITATION.

*Water analysis. Determination of hardness.* L. W. Winkler. Z. angew. Chem., 1921, 34, 115–116.

THE following modification of Wartba's method is recommended: 100 c.c. of water is treated with 2 drops of methyl orange solution and the "alkalinity" is titrated with N/10 hydrochloric acid. The mixture is then transferred to a 200 c.c. cylinder, 50 c.c. of N/10 sodium hydroxide-sodium carbonate solution is added, and the whole diluted to 200 c.c. After about 24 hrs., 100 c.c. of the clear solution is drawn off and the excess of alkali is titrated with N/10 hydrochloric acid.—W. P. S.

*Water analysis.* L. W. Winkler. Z. angew. Chem., 1921, 34, 143.

TO effect saving in the use of ethyl alcohol, soap solution for the determination of hardness in water may be prepared with propyl alcohol, the palmitic acid or oleic acid being dissolved in this solvent and neutralised with potassium hydroxide solution. Directions are also given for the determination of calcium and magnesium in waters.

—W. P. S.

*Phenol and phenol salt solutions; Methylene blue as an indicator of the toxicity of — towards yeast.* C. G. Fraser. J. Phys. Chem., 1921, 25, 1—9.

SOLUTIONS of phenol and of phenol and sodium chloride, which are in equilibrium with solutions of phenol in toluene or kerosene (*cf.* J., 1920, 799 A), are isotoxic with these towards yeast cells if ability to stain with methylene blue be adopted as a criterion of death. If, however, inability to form colonies on wort-agar be taken as indication of death, the solutions containing sodium chloride are more toxic. The inability to form colonies on wort-agar seems to mark an earlier stage in the loss of vitality, and the time it takes to reach this stage seems to depend on other factors than the temperature and chemical potential of the phenol in the solution, amongst them the osmotic pressure of the toxic liquid.—J. C. K.

*Phenol; Effect of alcohol on the toxicity of — toward yeast.* E. I. Fulmer. J. Phys. Chem., 1921, 25, 10—18. (*Of. supra.*)

YEAST cells derived from one original cell, and grown under the same conditions in wort, may show very different degrees of resistance to phenol. When wort is seeded with yeast, the first evidence of the fermentation is the formation of white flecks or foam on the surface. The time at which these flecks appear is quite definite. After this first stage, another is reached of active fermentation; while, in a third stage, the fermentation grows slower and finally ceases. When the alcohol produced by fermentation has reached a certain concentration, the cells are much more resistant to phenol than normal cells. If alcohol is added to the wort before seeding with yeast, the formation of abnormal "resting cells," which appear only in the third stage mentioned above, is hastened. By taking as a criterion of death the inability to grow colonies on wort-agar, it was found that a solution containing water, phenol, and 3.75% of alcohol is more toxic to normal yeast cells than a chemically equivalent solution without alcohol; but if, on the other hand, the ability to stain with methylene blue is taken as a criterion, the solutions are found to be equally toxic. Immersion in 8% solutions of alcohol in water increases the number of cells capable of staining with methylene blue, and, consistently with this, a solution containing phenol, water, and 7.5% of alcohol increases the percentage of stained cells more rapidly than a chemically equivalent solution of phenol in water.—J. C. K.

*Strawboard factory waste waters.* Hermann. See V.

#### PATENTS.

*Water; Apparatus for separating gases, iron, manganese, and other hardening admixtures from —.* H. Wehner. E.P. 7272, 14.5.15.

THE water in a finely divided state is mixed with air in a chamber under partial vacuum, the air serving to absorb deleterious gases and being then separated together with such gases. The admixture of the air and water is effected in a "diffuser," comprising a rotor having blades which work between fixed blades in a casing through which the water is drawn by suction, a suitable quantity of air being admitted through numerous small orifices in the circumference of the diffuser. From the diffuser the aerated water passes to a chamber under partial vacuum where the added air is again separated from the water, which is then drawn through a strainer (composed of slag or of a substance which, if desired, will effect the separation of iron etc.) by means of a centrifugal pump on the same shaft as the rotor of the diffuser, and delivered to a suitable tank. If desired

a pipe having a series of constrictions, or closely packed with balls, and provided with a porous cell through which air may be forced, may be interposed between the diffuser and the vacuum chamber.

—G. F. M.

*Water; Process for the sterilisation and purification of — [by means of decolorising carbon].* J. N. A. Sauer. E.P. 155,610, 20.6.19.

WATER is treated with finely divided decolorising carbon, such as Eponit or Norit, which has previously been subjected to the action of alkalis and/or acids and/or high temperatures; *e.g.*, the carbon may be heated with dilute sodium hydroxide or ammonia solution, then with dilute hydrochloric or other volatile acid, and finally heated alone to 1200° C. or above.—J. H. L.

*Water; Treatment of — for softening, sterilising, and like purposes.* H. J. Magrath. E.P. 158,498, 13.9.20.

THE plant consists of a tank divided into two parts, the lower of which contains a filtering medium, such as natural or artificial zeolite, and the upper a chamber for a regenerating solution with a removable filter and a ball cistern. The two parts of the tank are connected by pipes with a cock which can be set so as to pass either the water to be treated or the regenerating solution through the lower part. The plant is connected with the main water supply pipe in such a manner that it may be cut out of the circuit without disturbing any of the connexions. It is specially suitable for use in inhabited buildings. (*Of.* E.P. 18,867 of 1914; J., 1915, 1030.)

—J. H. J.

*Sterilising liquids [water]; Process and apparatus for — applicable also to like operations.* W. Paterson. E.P. 158,578, 1.8.17.

CHLORINE is passed through a measuring valve to an absorber to which a regulated amount of water is also admitted. The solution obtained accumulates in a reservoir, from which it passes to a feeding tank with a tapered outlet valve controlled by a float resting in the main body of water to be treated as it flows over a weir. In this way the flow of the main body of water determines the amount of solution to be added. When the accumulation of solution in the reservoir reaches a certain amount, the supply of gas and water is automatically cut off and is not resumed until the accumulation is reduced again.—J. H. J.

*Water; Process for the softening or purification of —.* B. F. Rushton. E.P. 158,620, 3.11.19.

A TANK with curved bottom contains the water to be treated. Electrodes of carbon, zinc, or iron are suspended from a bar across the top of the tank. In the lower portion of the tank is an agitator worked by an electric motor mounted above the tank. A current of about 25 volts is passed through the water for about 1 hr., after which the electrodes are removed, slaked lime is introduced in the proportion of 3 lb. to 1000 galls. of water, and the agitator is set in motion for 10 mins. The water is then allowed to stand for about 1 hr. and is run off through a valve in the lower portion of the tank. The sludge is removed from the bottom of the tank through a sluice valve. The process is suitable for the treatment of any mineral water except sea water.—J. H. J.

*Water; Purification of — by means of barium carbonate and quicklime.* H. Riesert G.m.b.H. G.P. 333,994, 25.1.18.

BARIUM carbonate is suspended in a rising column of water which, at the moment when the reaction between the barium carbonate and the sulphates in the water is complete, is treated with quicklime,

which tends to prevent the fine particles of barium sulphate from being carried over with the purified water.—A. R. P.

*Sewage and analogous liquids; Apparatus for the purification of —. Air distributing apparatus connected with the purification of sewage and analogous liquids.* W. T. Lockett and A. Herring-Shaw. E.P. (a) 156,816 and (b) 156,817, 11.8.17.

(a) An aerating device for the aeration of sewage consists of one or more revolving arms made in the form of a sector of a circle and projecting horizontally from a central pivot in a treatment tank. The arm is of hollow section and its upper surface is of porous material. It is supplied with compressed air from a central shaft or otherwise. The upper surface of the arm may be of a continuous flat form or may be composed of units of circular or other shape, graduated in size so as to give a sufficient air discharge at any position between the centre of rotation and the extremity of the arm. In another form, the arm consists of a pipe with a series of short branch pipes from its upper side terminating in flat circular air chambers with porous upper surfaces of graduated diameter according to their distance from the centre of rotation. (A) In order to lift deposited solids from the floor of the tank and bring them into the sphere of action of the air supply, the advancing side of the rotating arm is made with an inclined surface, sloping forwards from above downwards.—J. H. J.

*Waste water from gas-works and tanneries; Process for rendering innocuous —.* C. Bozenhardt. G.P. 333,703, 14.3.19.

AMMONIACAL waste water from gas-works is allowed to mix with the waste water from chrome leather tanneries containing salts of chromium, in order to recover the latter as chromium hydroxide and to render the resulting liquid safe for direct discharge into a river.—A. R. P.

*Insecticides, fungicides, animal dips; Materials for use as — and like purposes.* A. E. Hawker. E.P. 160,511, 15.12.19.

LIGHT wood oil, obtained by the redistillation of the tarry layer formed by the controlled distillation of hard woods at an initial temperature of 300° F. (about 150° C.), raised subsequently to 800° F. (about 430° C.), is used in combination with soap or other emulsifying agent, in tablet, powder, or liquid form, as an insecticide, fungicide, etc. The fraction of the distillate employed is preferably that collected up to the point at which the sp. gr. reaches about 0.98, but other fractions may be used if desired. A maximum of 50% of kieselguhr, fuller's earth, or other absorbent is used in making the powder form of the preparation, whilst water, glycerin, or other solvent is employed for the liquid form.—G. F. M.

*Sheep dips and cattle washes.* J. McDougall and F. Howles. E.P. 160,597, 7.1.20.

A FLUID sheep dip or cattle wash containing a high concentration of sodium arsenite is prepared by dissolving arsenious oxide in one-fourth or more of its weight of sodium hydroxide in concentrated aqueous solution, adding a colloid, such as hydrolysed starch or glue, casein, or soap, to the extent in the case of the hydrolysed starch of about one-twentieth of the weight of the arsenic, and evaporating the whole to a concentration of 50% or more of arsenious oxide. The mixture remains quite fluid on cooling and can be more easily diluted to the required strength for use than concentrated sodium arsenite preparations in the solid form.—G. F. M.

*Parasiticide for plants and animals.* Farbenfabr. vorm. F. Bayer und Co. G.P. 333,327, 4.7.19.

THE parasiticide consists of an ether containing at least one aralkyl group, such as dibenzyl ether, chlorobenzyl ethyl ether or xylyl phenyl ether. The ethers may be used in the liquid or vapour form, as pastes, absorbed in a porous powder, in soaps or ointments, or as solutions in alcohol or ethyl acetate, if necessary also in admixture with other substances.—A. R. P.

*Phenols; Preparation of solid mixtures [disinfectants] soluble in water and containing —.* Chem. Fabr. von Heyden, A.-G. G.P. 331,583, 23.3.13.

SOLID, insoluble or only slightly soluble complex compounds of phenols with alkali salts of the same or other phenols, are mixed with such solid salts of organic acids as yield aqueous solutions capable of dissolving phenols. The products dissolve readily in water and yield clear solutions possessing only slight alkalinity, and such alkalinity may be avoided by incorporating with the original mixture a solid acid or acid salt which forms with the free alkali a salt exerting a solvent action on the phenols.—J. H. L.

*Fatty matter from garbage.* U.S.P. 1,372,479. See XII.

*Fumigant paint.* U.S.P. 1,373,499. See XIII.

*Treatment of seeds.* G.P. 327,310. See XVI.

*Purifying liquors.* E.P. 155,609 and 155,611. See XVII.

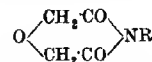
## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Antipyrine; Compounds of — with mercury.* E. Oliveri-Mandalà. Gazz. Chim. Ital., 1921, 51, I., 125—130.

ANTIPYRINE (1 mol.) combines readily with unstable mercury compounds (1 mol.), such as HgCl.OH, HgI.OH, and HgCl.NH<sub>2</sub>; the antipyrine apparently behaves as an unsaturated compound, the nitrogen atom joined to a methyl group uniting with the negative portion of the added molecule. Pyramidone (dimethylaminoantipyrine) combines with mercuric chloride, but not with Hg(OH)<sub>2</sub>, HgCl.OH, or HgCl.NH<sub>2</sub>, replacement of the methinic hydrogen of the antipyrine molecule by the dimethylamino-group present in pyramidone evidently destroying the mobility of arrangement of the valencies of the nucleus. (Cf. J.C.S., May.)—T. H. P.

*Sweetening materials; Cyclic imide ethers of diglycollic acid as —.* M. Sido. Ber. deuts. Pharm. Ges., 1921, 31, 118—129.

WHILST the substitution of the imino hydrogen in saccharin by alkyl groups entirely destroys the sweetness of that substance, the position is completely reversed in the case of diglycolimide, which, though itself tasteless, yields alkylimides of the general formula



increasing in sweetness with an increasing number of carbon atoms in the alkyl group up to a maximum in the N-propyl derivative. The aryl derivatives on the other hand are almost tasteless. The substituted imides were prepared by heating the acid alkylamine or arylamine diglycolates in a vacuum, whereby water was expelled, and the residual imide was purified by distillation under reduced pressure



and crystallisation if necessary from absolute alcohol. Diglycolmethylimide and diglycolethylimide are solid crystalline substances, whilst the *N*-propyl- and *N*-butylimides are colourless, oily liquids having an intensely sweet taste. All four substances are extremely hygroscopic, and as they are rapidly reconverted by the action of water into the tasteless alkylamine hydrogen diglycollates, they can have no practical application as sweetening agents. (Cf. J.C.S., June.)—G. F. M.

**Catalysts; Influencing of the activity of — II.**  
*Reduction of acid chlorides to alcohol and ester.*  
K. W. Rosenmund, F. Zetzsche, and F. Heise. Ber., 1921, 54, 638—647. (Cf. J., 1921, 321 A.)

THE reduction of benzoyl chloride with the aid of uninfluenced catalysts leads mainly to the production of hydrocarbons. In the presence of sulphur-nitrogen compounds, aldehydes are formed. The joint influence of toluene and xanthone causes benzyl benzoate to be the main product. In xylene solution, benzyl esters undergo reductive fission into alcohol and toluene, which is, however, inhibited by addition of the latter. Quinoline favours the production of benzyl alcohol, dibenzyl ether being formed simultaneously. (Cf. J.C.S., June.)—H. W.

**Glyoxal; Preparation of — by the action of acetylene on gold chloride or bromide.** K. Kindler. Ber., 1921, 54, 647—649.

GOLD is precipitated quantitatively from aqueous solutions of auric halides by acetylene if the gold content does not greatly exceed 1.5%. The acetylene is converted into carbon dioxide (12%) and glyoxal (about 86%), the latter appearing to be the sole organic product of the change. The finely-divided gold can be readily re-converted into the halide by treatment with chlorine or bromine.—H. W.

**Oxalic acid; Volumetric determination of —.**  
A. Abelmann. Ber. deut. Pharm. Ges., 1921, 31, 130—131.

TO the oxalic acid or oxalate solution in a 100 c.c. flask, 30—40 drops of 5*N* nitric acid, and a measured excess of *N*/10 mercuric nitrate solution, containing sufficient nitric acid to produce a clear solution, are added, followed by about 50 c.c. of saturated potassium nitrate solution and sufficient water to make 100 c.c. After standing for 15 mins., the solution is filtered and an aliquot portion of the filtrate titrated with *N*/10 ammonium thiocyanate with ferric ammonium sulphate as indicator.

—G. F. M.

**Lactic acid; Detection of —.** L. Hartwig and R. Saar. Chem.-Zeit., 1921, 45, 322.

ABOUT 0.2 c.c. of a solution containing not more than 0.2% of lactic acid is mixed with 2 c.c. of concentrated sulphuric acid, the mixture heated at 100° C. for 2 mins., cooled, and treated with 2 drops of 5% alcoholic guaiacol solution; a red coloration is obtained. Formic, acetic, malic, benzoic, and salicylic acids do not give a coloration with the test; citric acid yields a yellow, tartaric acid a faint pink, and tannin a blackish-violet coloration. To detect lactic acid in baking powder, the sample is moistened with water, acidified with phosphoric acid, then mixed with sand and plaster of Paris, and the mixture extracted with ether; the residue obtained on evaporating the ethereal solution is diluted with water and tested as described.

—W. P. S.

**Calcium salt; A new —.** L. Gaucher and G. Rollin. Comptes rend. Soc. Biol., 1921, 84, 303—304. Chem. Zentr., 1921, 92, I., 562.

By the action of phosphorus tri-iodide on concentrated lactic acid a substance of the formula,

$C_4H_4O_4P_3$  is obtained. It melts at 120° C., and is converted by water into the tribasic dipropanolol-phosphoric acid,  $HO.P(O.CH(CH_2).CO_2H)_3$ . The calcium salt of this acid,  $Ca_3(C_4H_4O_4P_3)_2.8H_2O$ , is very suitable for subcutaneous injection. In neutral or weak acid solution it remains unchanged in the cold, but in presence of alkali it is converted into insoluble calcium phosphite.—G. F. M.

**Acetone; Action of ammonia on —.** T. S. Patterson and A. McMillan. Chem. Soc. Trans., 1921, 119, 269—271.

IN the course of the preparation of diacetoneamine from acetone and ammonia by the usual method, what appears to be an intermediate product of the constitution,

$CH_3.CO.CH_2.C(CH_3)_2.NH.C(CH_3)_2.NH_2$ , was deposited in thin prismatic crystals when the ammoniacal acetone was cooled in a freezing mixture. The substance melts at 45° C., and behaves as a di-acid base when titrated with hydrochloric acid in aqueous solution. A monohydrochloride is precipitated when the ethereal solution is treated with dry hydrogen chloride. The substance readily decomposes, and it is suggested that when the reaction mixture is worked up in the usual way it is decomposed by the treatment with oxalic acid into ammonium oxalate and diacetoneamine hydrogen oxalate.—G. F. M.

**Camphor; Reaction for distinguishing natural and artificial —.** Utz. Farben-Zeit., 1921, 26, 1065.

LENZ's hydrochloric acid reaction (J., 1911, 766) for distinguishing between natural and artificial camphor is untrustworthy, whilst the colour reactions with fuming nitric acid and sulphuric acid observed by Zimmermann (Apoth.-Zeit., 1920, 35, 382) were most probably due to fortuitous impurities. The author recommends the vanillin-hydrochloric acid and vanillin-hydrochloric-sulphuric acid tests for distinguishing between natural and synthetic camphor (cf. Bohrisch, J., 1915, 1224). In cases where determinations of synthetic camphor in natural camphor are required, polarimetric examination should be resorted to.

—A. de W.

#### PATENTS.

**Valeric acid and alkaline valerates; Manufacture of —.** Darrasse Frères et Cie., and L. Dupont. E.P. 137,064, 24.12.19. Conv., 3.12.18.

SALTS of valeric acid are obtained by heating an excess of amyl alcohol with sodium or potassium hydroxide in an autoclave provided with an adjustable discharge valve for the escape of hydrogen at 235°—240° C. under 18 atm. pressure. When the whole of the alkali has been converted into valerate according to the equation  $C_5H_{11}O + NaOH = C_5H_9O_2Na + 2H_2$ , the excess of amyl alcohol is distilled off in a current of steam, and valeric acid is isolated from the resulting alkali valerate solution by the usual methods.—G. F. M.

**Acetaldehyde from acetylene; Process for the manufacture of —.** Chem. Fahr. Griesheim-Elektron. E.P. 143,891, 27.5.20. Conv., 10.8.16.

IN the conversion of acetylene into acetaldehyde in presence of mercury compounds the catalyst is continuously regenerated in the absorption vessel itself by an anodic oxidation process in which the reduced mercury forms the anode, the acid reaction liquid the electrolyte, and mercury, lead, or platinum the cathode. The cathode may either be arranged in a chamber closed by a diaphragm from which the hydrogen is discharged, or it may be placed without a diaphragm in the acid reaction liquid, the hydrogen which gradually accumulates in the circulating gas being removed at intervals. By this process larger quantities of mercury salt,



exceeding 10% of the weight of the acid reaction liquid, can be employed, with a consequent increase in the rate of absorption of the acetylene.

—G. F. M.

*Borneol; Manufacture of* —. Fabr. de Prod. Chim. de Thann et de Mulhouse. E.P. 144,604, 8.12.19. Conv., 12.6.19.

TURPENTINE or crude pinene is heated with a quarter of its weight of tetrachlorophthalic acid in a reflux apparatus with continual stirring for 12 hrs. at 106°–108° C. The temperature is then slowly raised to 140° C., at which it is maintained for 6 hrs. After cooling, the unattacked terpenes are distilled off with steam or in a vacuum, and the residue, which forms a glassy mass consisting of dibornyl tetrachlorophthalate, is hydrolysed with alcoholic sodium hydroxide. The borneol, precipitated by the addition of water, is purified by the usual methods. It is optically active, and, unlike the product obtained by the action of other organic acids on pinene, it is quite free from isoborneol. The yield may be as high as 18% of the crude pinene used, and in addition there is a considerable quantity of pinene recovered, which, if desired, can be returned to the manufacture. The tetrachlorophthalic acid can be almost quantitatively recovered.—G. F. M.

*Isobornyl ester and camphene; Conversion of pinene compounds into a mixture of* —. L. G. Wesson. U.S.P. 1,372,382, 22.3.21. Appl., 30.6.19.

PINENE hydrochloride or hydrobromide when heated with a carboxylic acid in presence of not more than 5% of zinc is converted into a mixture of an isobornyl ester and camphene.—G. F. M.

*p-Aminophenol and of its O-alkyl ethers; Manufacture of derivatives of* —. E. Kolshorn. E.P. (A) 145,614, 29.6.20. Conv., 13.6.19. (B) 155,575, (C) 155,576, 24.11.20. Conv., 12.12.19.

(A) WATER-SOLUBLE derivatives of *p*-aminophenol or of its O-alkyl ethers are obtained by their conversion into *N*-dihydroxypropyl compounds of the general formula,



by causing the amino-compounds to react either directly or in a neutral solvent with monochlorhydrin, or with epichlorhydrin alcohol, in the former case either with or without the addition of an alkali hydroxide. Thus 22 gr. of *p*-aminophenol when gently heated with 15 g. of epichlorhydrin alcohol (glycide) in benzene solution is converted into *N*-dihydroxypropyl-*p*-aminophenol, m.p. 192° C., readily soluble in water, giving a solution which has a powerful reducing action on silver salts, and is specially suitable as a photographic developer. *N*-Dihydroxypropyl-*p*-phenetidine, m.p. 93° C., is obtained by the action of  $\alpha$ -monochlorhydrin (11 g.) on *p*-phenetidine (14 g.) in presence of alcoholic potassium hydroxide. It is easily soluble in water and has valuable therapeutic properties. (a) The same derivatives of *p*-aminophenol and its ethers are obtained by condensing the bases with epichlorhydrin, with or without the addition of a neutral solvent, and treating the chloro-compound,  $\text{RO.C}_6\text{H}_4.\text{NH.CH}_2.\text{CH(OH).CH}_2\text{Cl}$ , produced, with the theoretical amount of alcoholic potassium hydroxide. (c) Instead of epichlorhydrin,  $\alpha$ - or  $\beta$ -dichlorhydrin may be used in presence of pyridine or alcoholic potassium hydroxide to eliminate the halogen acid, the chloro-intermediate product being treated with alkali hydroxide as before to eliminate chlorine.—G. F. M.

*Glandular extractive product [from the infundibular lobe of the pituitary gland]; Process of manufacturing* —. A. M. Clover, Assr. to Parke, Davis and Co. U.S.P. 1,373,551, 5.4.21. Appl., 9.3.13.

THE gland is extracted with hot, acidified water,

insoluble material is separated from the solution, and the active constituent is precipitated by saturating the solution with sodium chloride.

—L. A. C.

*Trihydroxyisopentane; Preparation of* —. Farbenfabr. vorm. F. Bayer und Co. G.P. 309,111, 12.8.17.

TRIHYDROXYISOPENTANE (dimethylglycerol) is obtained by the oxidation of 3-methylbutenol, or by the saponification of the corresponding chlorine derivative. It is a solid, m.p. 49° C., b.p. 123° C., and is more hygroscopic and more strongly bactericidal than glycerin.—B. V. S.

*Anti-gonorrhœa preparation; Production of* —. P. Bergell. G.P. 330,348, 1.4.19.

A CULTURE of gonococci is prepared under optimum conditions at 36° C. and then the organisms are killed by maintaining a temperature of 41°–42° C. for a long time. The destroyed organisms are suspended in physiological salt solution with the addition, if necessary, of a disinfectant. If the temperature is raised to 45° C. the anti-toxic properties of the culture are reduced.—H. J. H.

*Ovaries and similar organs; Preparation of nitrogenous compounds from* —. L. Seitz and H. Wintz. G.P. 332,165, 25.1.16. Addn. to 320,857.

THE method of the chief patent (J., 1920, 675 A), is extended to the ovary and the placenta, the former yielding a substance with a preventive effect and the latter one with a loosening effect on menstruation. The latter substance may also be obtained in pure form from the dried, powdered organ by extraction with alcohol and chloroform.—B. V. S.

*Glycerin substitute [from lactates]. Chem. Fabr. vorm. Goldenberg, Geromont und Co. G.P. 332,167, 9.8.18. Addn. to 303,991 (J., 1920, 756 A).*

MIXTURES of alkali lactates with lactates of suitable di- and trivalent metals are employed. Lactates of calcium, zinc, and magnesium obtained in the manufacture of lactic acid may thus be utilised.

—J. H. L.

*N-Alkyl-acylhomopiperonylamines; Manufacture of* —. E. Merck, Chem. Fabr. G.P. 332,474, 26.3.12.

THE alkali salts of *N*-acylhomopiperonylamines are treated with alkylating agents. For example, by condensing the potassium compound of formylhomopiperonylamine (obtained by treatment with potassium in toluene solution) with methyl iodide, methylformylhomopiperonylamine, a thick, strongly refracting oil, b.p. 194° C. at 6 mm., is formed quantitatively. The sodium compound prepared in a benzene solution may be allowed to react in a similar manner on methyl chloride.—C. A. C.

*Fatty acids and aldehydes; Manufacture of* —.

C. Harries, R. Koetschau, and E. Albrecht. G.P. 332,478, 11.6.16. Addn. to 324,663 (J. 1920, 765 A).

THE decomposition of the ozonides is effected by reducing agents, as sulphur dioxide, sulphites, zinc or the like and acids. The dark-coloured soaps and acids obtained by alkali fusion of hydrocarbons (G.P. 314,745; J., 1920, 151 A) may also be transformed into ozonides and the latter decomposed by steam, caustic alkali, or sulphuric acid, or by reducing agents, as sulphur dioxide, sulphites, zinc, and acids. Technically valuable light yellow to colourless products of low molecular weight are formed, consisting chiefly of carboxylic and dicarboxylic acids. During the action of ozone on the dark, alkaline solutions, e.g. of acid resins from the re-

fining of petroleum, the ozonides are decomposed *in statu nascendi*.—C. A. C.

**Bismuth-alkali iodide solutions; Preparation of — for therapeutic use.** Chem. Fabr. auf Aktien (vorm. E. Schering). G.P. 332,552, 21.4.16.

THE capacity to precipitate albumin or gelatin from solution is removed by the addition to the bismuth iodide of a large excess of an alkali iodide and a small quantity of an acid such as tartaric, glycollic, citric, lactic, or hydriodic acid. The solution may contain, for example, 0.5 g. of sodium-bismuth iodide, 7.5 g. of sodium iodide, and 1 g. of tartaric acid in 500 c.c. of water. The solutions have a strong germicidal action.—B. V. S.

**Chloral and a phenol; Manufacture of a compound of —.** O. Hinsberg. G.P. 332,678, 15.4.17.

By the interaction of chloral and *p*-acetaminophenol, with or without a solvent, chloral-acetaminophenol,  $\text{CH}_3\text{CO.NH.C}_6\text{H}_4\text{O.CH(OH).CCl}_3$ , is obtained. It forms a colourless and tasteless crystalline powder, m.p. about 180° C. When heated quickly it is decomposed with frothing and evolution of chloral vapour. Chloral is also liberated on boiling with water. The product acts as a soporific in smaller doses than chloral, and its action also differs appreciably from that of the latter.—C. A. C.

**Glycol diacetate; Manufacture of —.** K. H. Meyer. G.P. 332,677, 29.7.19.

ETHYLENE DICHLORIDE and anhydrous sodium acetate are heated in presence of glycol diacetate. At 230° C. a smooth conversion takes place which would only be effected in a small degree without a solvent. Glycol diacetate, like the monoacetate, is a good solvent and gelatinising agent.—C. A. C.

**Urea; Manufacture of — from carbonic acid compounds of ammonia.** Badische Anilin- und Soda-Fabr. G.P. 332,679, 11.7.15.

A "MELT" of ammonium carbamate, or of the product of the interaction of carbon dioxide and ammonia together with a small amount of water, is prepared in a separate vessel and is then forced under pressure into a reaction vessel. By making the reaction vessel part of a pressure-proof system the process may be made continuous. For example, a "melt" of 10 pts. of ammonium carbamate and 1 pt. of water, prepared at 40° C. under a pressure of 15 atm., is passed through a heated pressure-proof spiral pipe, heated at 135°–140° C. If the time occupied in passing through the spiral pipe is 2 to 3 hrs., then on relieving the pressure 25% of the carbamate will have been converted into urea.—C. A. C.

**Urea melts obtained from carbon dioxide compounds of ammonia; Treatment of —.** Badische Anilin- und Soda-Fabr. G.P. 332,680, 11.7.15.

THE ammonium salts which have not been converted are distilled under pressure from the urea autoclave into another pressure-proof vessel at a lower temperature. The greater part of the ammonium carbamate and carbonate is easily and quickly precipitated in a solid or molten condition respectively. The pressure-proof receiver may either be a second urea autoclave or with advantage the vessel used for preparing the melt (*cf. supra*). The ammonium salts remaining in the urea autoclave are afterwards distilled off under normal or reduced pressure.—C. A. C.

**Guanidine salts; Manufacture of —.** F. Hoffmiller. G.P. 332,681, 12.9.17.

ALKALINE-EARTH salts of cyanamide are heated together with ammonium salts to a suitable temperature; e.g., calcium cyanamide is added to a melt of

ammonium nitrate and then heated to 200°–220° C. The following reaction takes place:  $\text{CaCN}_2 + 3\text{NH}_4\text{NO}_3 = \text{C}(\text{NH})_2(\text{NH}_2)_2 + \text{HNO}_3 + \text{Ca}(\text{NO}_3)_2 + 2\text{NH}_3$ . By solution, filtration, and crystallisation carbon, calcium nitrate, and guanidine nitrate are separated. The calcium nitrate remaining in the mother liquors is worked up to ammonium nitrate by means of the ammonia obtained.—C. A. C.

**Aldehydes; Preparation of —.** K. W. Rosenmund. G.P. 333,154, 28.3.17.

ACID halides, or solutions of the same, are treated with hydrogen in the presence of a catalyst and a substance capable of combining with acid. Stearic aldehyde is prepared from a solution of stearic acid chloride in benzol in the presence of 5% of palladinised barium sulphate and calcium carbonate; benzaldehyde, *p*-hydroxybenzaldehyde, and butyraldehyde are prepared respectively from benzoyl bromide (or chloride), *p*-carbomethoxybenzoyl chloride, and butyryl chloride, using palladinised silicic acid as catalyst.—L. A. C.

**Urea or ammonia; Production of — from [calcium] cyanamide.** S. Giertsen. E.P. 160,857, 14.8.19.

SEE U.S.P. 1,326,045 of 1919; J., 1920, 174 A.

**Denitrating nitric esters.** G.P. 333,708. See XXII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Photographic emulsion; Orientation of the grains in a dried —.** L. Silberstein. J. Opt. Soc. Amer., 1921, 5, 171–177.

A MATHEMATICAL analysis is given of the probable average orientation of silver halide grains in a sensitive film, making certain simple assumptions as to the nature of the drying process. It would appear highly probable that the "efficiency coefficient," i.e., the ratio between the average projected area of the grain and its actual area, regarding all the grains as flat plates of small thickness, is very nearly unity in the dried film.—B. V. S.

**[Photographic] reduction of developing-out papers; Sensitometric study of —.** L. A. Jones and C. E. Fawkes. J. Franklin Inst., 1921, 191, 503–516.

IN testing the effects of photographic reducers on developing-out papers, graded prints, obtained in the usual way by exposure in a non-intermittent sensitometer, and development, were measured by means of a reflectometer, then reduced, and again measured. A comparison of the curves obtained from these measurements shows both the extent and the nature of the reducing effect. Ten reducers were tested, full particulars being given of six of them, divided into 3 types. Potassium permanganate solution and Belitaki's reducer (ferric chloride, sodium oxalate and thiosulphate) are of type 1, the contrast of the print being unaltered. Type 2, in which contrast is increased by reason of more vigorous action on the lower densities, includes iodine-cyanide and Farmer's (ferricyanide-thiosulphate) reducers. The third type, in which contrast is reduced, includes ammonium persulphate and the Nietz-Huse proportional reducer (permanganate-persulphate).—B. V. S.

### PATENTS.

**Colour photography.** W. Friese-Greene, J. N. Thomson, and Colour Photography, Ltd. E.P. 160,540, 20.12.19 and 15.4.20.

A TONING solution, chiefly intended for production of the red constituent in two- or three-colour photo

graphy, contains potassium ferricyanide, uranium nitrate, Rose Bengale, Naphthol Yellow, and acetic acid along with iodine and/or Sensitol Red (Pincyanol).—B. V. S.

*Photographic developers.* J. Hauff und Co. G.P. 327,111, 24.10.18, and 328,617, 10.12.18.

THE carboxylic and sulphonic acids of *o*- and *p*-aminophenol, of *o,p*-diaminophenol and of their homologues, *e.g.*, *p*-aminosalicylic acid, are used in caustic alkaline solution. The developers show no tendency to the formation of veil. The resulting silver image is brownish-black in colour.—B. V. S.

*Photographic flash light; Production of* —. K. Hempel. G.P. 330,531, 15.10.19.

METALLIC aluminium and magnesium, together or singly, are mixed with manganite and/or permanganate of barium or mixtures of these, together with paraffin wax. Production of smoke is considerable owing to the absence of potassium.

—H. J. H.

*Photographic silver films; Intensifying* —. H. Franke. G.P. 333,094, 17.4.20.

By the use of solutions of selenium compounds, such as a selenosulphate, selenium is deposited on the silver grains. It may be removed again by treatment with, *e.g.*, permanganate solution slightly acidified with hydrochloric acid.—B. V. S.

*Coloured photographic pictures; Method and apparatus for producing* —. J. H. Christensen. U.S.P. 1,373,053, 29.3.21. Appl., 2.8.18.

SEE G.P. 313,836 of 1918; J., 1920, 45 A.

*p-Aminophenol derivatives.* E.P. 145,614 and 155,575-6. See XX.

## XXII.—EXPLOSIVES; MATCHES.

*Mercury fulminate; Characteristic reaction for the detection of* —. A. Langhans. Z. anal. Chem., 1921, 60, 93-94.

If mercury fulminate is moistened with alcohol, water added, and the mixture shaken with 20% sodium sulphantimonate solution, a yellow precipitate forms which gradually turns olive-green and then black. The filtrate from the precipitate gives a bright red colour with nitric acid; the colour is soluble in ether.—W. P. S.

### PATENTS.

*Nitroglycerin; Process for separating — from wash-waters.* Westfälisch-Anhaltische Sprengstoff A.-G. G.P. (a) 299,030, 5.11.15, and (b) 299,720, 5.2.16.

(a) BOTH suspended and dissolved nitroglycerin may be removed from the wash-waters by treating them with nitrocellulose. (b) Nitroglycerin is separated from a solution in aqueous acetone, containing up to 30% of acetone, by shaking the solution with, or filtering, it through, nitrocellulose. The process should be conducted at a low temperature in order to minimise loss of acetone.—W. J. W.

*Smokeless powders; Process for comminuting* —. Westfälisch-Anhaltische Sprengstoff A.-G. G.P. 332,284, 25.1.19.

POWDERS with a hard grain or surface are softened by means of gelatinising agents, or preferably, aqueous volatile solvents, and are then pulverised with addition of water in kneading machines.

—W. J. W.

*Nitric acid esters of carbohydrates and glycerol; Process for completely or partially denitrating* —. H. Bucherer. G.P. 333,708, 14.12.18.

THE ester, *e.g.*, nitrocellulose, is mixed with or dissolved in sulphuric acid, and an aromatic compound, such as benzene, is added with vigorous agitation. The nitro groups migrate to the aromatic compound and the product settles into separate layers of benzene and sulphuric acid containing respectively nitrobenzene and cellulose in solution. Sulphonic acids or acid sulphates can be employed instead of sulphuric acid.—L. A. C.

## XXIII.—ANALYSIS.

*Melting points; Determination of* — [especially of inorganic substances]. [Melting point of potassium chlorate.] C. D. Carpenter. Chem. and Met. Eng., 1921, 24, 569-571.

THE apparatus employed consists of a lagged beaker, with observation holes in the lagging, a platinum resistance thermometer, and a stirrer of which the speed can be adjusted. For the heating bath a mixture of potassium and sodium nitrates (m.p. 220° C.) is used. For temperatures above 600° C. both bath and melting point tube must be constructed of transparent quartz. In determining the melting point of potassium chlorate, the substance was melted and then cooled, and this process was repeated several times, the temperature being kept within narrow limits, and the crystals never being allowed to disappear completely. The temperatures at which new crystals formed on cooling were noted in terms of the resistance. By plotting the results on a graph, the resistance was found to be 116·435 ohms, corresponding to 357·10° C.

—W. J. W.

*Sodium sulphantimonate; Reactions of — with some metallic salts.* A. Langhans. Z. anal. Chem., 1921, 60, 91-93.

THE coloration of the precipitates obtained when metallic salt solutions are treated with freshly-prepared sodium sulphantimonate solution is as follows: Iron, nickel, cobalt, and bismuth, black; aluminium, chromium, silver, copper, and lead, brown; manganese, orange; zinc and cadmium, yellow. Mercuric oxide is coloured black by the reagent, as are also mercuric oxalate and mercurous nitrate, whilst mercuric bromide is coloured yellow. Mercuric cyanide is at first coloured yellow and then black; mercuric chloride yields a reddish-yellow compound. Sodium stannate and arsenate do not give reactions. (See also Cl. XXII.)

—W. P. S.

*Adsorption; Importance of — in analytical chemistry. IX. Glass wool as a filter material.* I. M. Kolthoff. Pharm. Weekblad, 1921, 58, 463-471.

GLASS-WOOL should not be used for filtering warm dilute solutions of salts of metals or alkalis, as considerable losses occur. (Cf. J.C.S., June.)

—S. I. L.

See also pages (a) 343, *Lignified cell membranes* (Casparis); *Sulphite and sulphate pulps* (Lofton and Merritt). 345, *Nitric acid in presence of nitrous acid* (Oliveri-Mandalà). 350, *Carbon in steel and iron* (Burkardt). 355, *Hexabromide value of oils* (Eibner); *Soap stock* (Fahrión). 356, *Acidity of ink* (Mitchell). 357, *Varnishes* (Wolff). 359, *Soils* (Hissink). 361, *Sucrose* (Jackson and Gillis); *Sugars* (Baker and Hulton); *Determination of copper and its use in sugar analysis* (Shaffer and Hartmann). 363, *Iron in wines* (Mathien). 365, *Water analysis* (Winkler). 368, *Oxalic acid* (Abelmann); *Lactic acid* (Hartwig and Saar); *Camphor* (Utz). 371, *Mercury fulminate* (Langhans).

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Adams, Adams, and Linkie. Mixing-machines. 13,051. May 7.  
Beldimano, Robertson, and Stanley. Evaporating or heating liquids. 13,082. May 7.  
Blair, Campbell, and McLean, Ltd., and Wright. Apparatus for effecting intimate contact of liquids and gases. 13,004. May 6.  
Buxton. Drying systems. 12,497. May 2.  
Curtis and Jones. Drying apparatus. 13,045. May 7.  
Evans (Allgem. Elektrizitäts-Ges.). Apparatus for effecting perfect combustion of coal, coke, etc. in furnace plants. 12,394. Apr. 29.  
Frischer. Apparatus for carrying out chemical and physical processes. 12,984. May 6.  
Jewell. Stills. 12,220. Apr. 28.  
Kenyon and Reynolds. Separating or grading powdered or granular materials, and treatment thereof by gases etc. 11,860. Apr. 25.  
Paterson. Filtering-apparatus. 11,972. Apr. 26.  
Pessi. Preventing incrustation in steam boilers. 12,981. May 6. (Ital., 6.5.20.)  
Roberts. Grinding-mills. 11,876. Apr. 25.  
Sklenar. Reverberatory furnace. 12,078. Apr. 27.  
Thompson (Sharples Separator Co.). Centrifugal emulsifiers. 12,048. Apr. 27.  
Thorne. 12,433. See IX.

#### COMPLETE SPECIFICATIONS ACCEPTED.

11,142 (1919). Miller, and Fletcher and Co. Filters. (161,993.) May 4.  
22,661 (1919). Philip. Stills. (162,000.) May 4.  
32,408 (1919). Patrick, Lovelace, and Miller. Separating and recovering gases. (137,284.) May 4.  
2703 (1920). Meston. Separating suspended matter from liquids. (162,390.) May 11.  
6962 (1920). Malkin. Grinding-mills. (162,481.) May 11.  
7182 (1920). Soc. l'Air Liquide. Protection for walls of enclosures in which reactions take place at high temperatures and pressures. (140,083.) May 11.  
9432 (1920). Holmes and Co., Henshaw, and Whittell. Apparatus for bringing liquids and gases into intimate contact. (162,166.) May 4.  
10,852 (1920). Soc. des Condenseurs Delas. Cooling viscous liquids. (142,454.) May 4.  
15,019 (1920). Rambaud. Evaporating and concentrating apparatus for syrups etc. (144,631.) May 11.  
15,182 (1920). Petree. Apparatus for separating impurities in suspension from liquids. (162,206.) May 4.  
20,214 (1920). Petzel. Separation of gas mixtures. (148,302.) May 11.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Amend. Refining petroleum distillates. 12,461. Apr. 30.  
Bamber and Parker. Gas producers or generators. 12,789. May 4.  
Bismarckhütte. Coking-chambers for generators. 12,974. May 6. (Ger., 6.5.20.)  
Cotton. Fuel. 12,951. May 6.  
Clelow. Disintegrating, dehydrating, etc. peat. 11,828. Apr. 25.

Duckham, and Woodall, Duckham, and Jones. Carbonising fuel in vertical retorts. 12,675. May 3.  
Evaos (Allgem. Elektrizitäts Ges.). 12,394. See I.  
Fournier. Manufacture of artificial fuel. 12,768. May 4. (Fr., 10.5.20.)  
Jacobs. Drying lignite, peat, turf, etc. 12,911. May 5. (Ger., 27.5.20.)  
Kirke. Water-gas plant. 12,889. May 5.  
Kratochwill. Artificial fuel. 12,670. May 3.  
Lloyd. Manufacture of coal etc. briquettes. 12,808. May 4.  
Midland Coal Products, Ltd., Fisher, and Montgomery. Apparatus for distilling coal etc. 12,950. May 6.  
Ricardo. Fuel for internal-combustion engines. 11,953. Apr. 26.  
Salerni. Apparatus for distilling carbonaceous materials. 11,838. Apr. 25.  
Soc. du Gaz de Paris. Manufacture of illuminating gas. 12,260. Apr. 28. (Fr., 18.5.20.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

26,589 (1919). Pease. See VII.  
32,165 (1919). Diver. Recovery of oil from bitumen, shale, etc. *in situ*. (162,337.) Apr. 11.  
1512 (1920). Marr, and Coke Oven Construction Co. Coke ovens. (162,045.) May 4.  
1576 (1920). International Coal Products Corp. Carbonising lignites. (142,443.) May 4.  
1846 (1920). Hudson. Making decolorising carbon. (139,156.) May 4.  
2316, 2320, 2322-4, 2326 (1920). American Coke and Chemical Co. Coke ovens etc. (138,124, 138,126, 138,128, 138,333-5.) May 11.  
2333 (1920). Watchman Co., and Morris. Gas-generating apparatus. (162,085.) May 4.  
3698 (1920). De Bruyn, Ltd., and Revis. Production of decolorising charcoal. (162,117.) May 4.  
3863 (1920). Drakes, Ltd., and Drake. Vertical retort settings. (162,422.) May 11.  
5247 (1920). Williams and Williams. Gas-producer. (162,455.) May 11.  
5403 (1920). Nielsen and Garrow. Continuous production of solid and gaseous fuel with by-product recovery. (162,459.) May 11.  
5666 (1920). Perry. Apparatus for distilling carbonaceous material. (162,136.) May 4.  
8256 (1920). Bean. Producing gas. (162,159.) May 4.  
14,106 (1920). Rambush. Removal of sulphur from gases. (162,554.) May 11.  
24,471 (1920). Krupp A.-G. Separating slags containing iron from coke and other fuel residues. (152,642.) May 4.  
29,412 (1920). Marr, and Coke Oven Construction Co. Coke ovens. (162,236.) May 4.  
30,947 (1920). Gewerkschaft Emscher-Lippe, and Heyn. Coking-plants. (153,313.) May 11.

### III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Barrett Co. Manufacture of products of oxidation of aromatic hydrocarbons. 12,153. Apr. 27. (U.S., 18.6.20.)  
Bismarckhütte. Separating water from coal tar. 12,974. May 6. (Ger., 6.5.20.)  
Bradley, Glossop, and Willsdon. Distillation of tar etc. 12,524. May 2.  
Commin. Treatment of pitch. 12,388. Apr. 29.

#### COMPLETE SPECIFICATION ACCEPTED.

20,585 (1920). Sudfeldt u. Co. Obtaining salts of sulpho acids from lignite tar oils. (148,763.) May 11.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

Dreyfus. Manufacture of cellulose derivatives. 11,854. Apr. 25.

Forster. Obtaining transparent effects on cotton fabrics. 12,463. Apr. 30. (Ger., 30.4.20.)  
McRae. Treatment of bamboo, bagasse, etc. for extraction of cellulose. 12,919. May 5.  
Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of cellulose compounds. 12,190. Apr. 28.  
Soc. Pichard Frères. Process for carotting hairs. 12,259. Apr. 28. (Fr., 15.5.20.)

## COMPLETE SPECIFICATIONS ACCEPTED.

24,519 (1919). White and White. Wool-washing machines. (162,313.) May 11.  
2721 (1920). Great Northern Paper Co. Paper-making. (141,022.) May 4.  
27,169 (1920). Great Northern Paper Co. Paper-making. (151,623.) May 4.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

## APPLICATIONS.

Crowther. Machines for treating textile fibres with liquids. 13,061. May 7.  
Ferguson and Rhodes. Treatment and dyeing of woollen yarns etc. 12,713. May 4.

## COMPLETE SPECIFICATION ACCEPTED.

13,731 (1920). Kershaw. Bleaching of fabrics. (162,195.) May 4.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

## APPLICATIONS.

Aluminium-Industrie A.-G. Manufacture of nitrate of calcium. 12,901. May 5. (Switz., 19.5.20.)  
Boorman and Browning. 12,157. See XVI.  
Brightmore. Fixation of atmospheric nitrogen. 12,146. Apr. 27.  
Chem. Fabr. Griesheim-Elektron, and Suchy. Electrolytic production of potassium carbonate from potassium chloride solutions. 12,573. May 2.  
Constant and Raisin. Production of boron. 11,947. Apr. 26. (Fr., 30.4.20.)  
Heenan and Froude, Ltd., and Walker. Concentration of brine. 12,600. May 3.  
Norris. Recovering sodium sulphate and a liquor of high acidity from nitre-cake. 13,029. May 7.  
Parrish, and South Metropolitan Gas Co. Manufacture of neutral sulphate of ammonia. 12,116. Apr. 27.

## COMPLETE SPECIFICATIONS ACCEPTED.

17,624 (1917). Buchner. Production of aluminium hydroxide. (162,303.) May 11.  
26,589 (1919). Pease. Extraction of ammonia from gases. (162,314.) May 11.  
31,936 (1919). Field, and Metals Extraction Corp. Purification of zinc solutions. (162,030.) May 4.  
1240 (1920). Ellis. See X.  
2325 (1920). American Coke and Chemical Co. Ammonia saturators. (138,129.) May 11.

## VIII.—GLASS; CERAMICS.

## APPLICATIONS.

Stafford. Glass etc. furnaces. 11,893-4. Apr. 25.  
Travers. Glass furnaces. 12,673. May 3.  
COMPLETE SPECIFICATIONS ACCEPTED.  
8949 (1920). Thompson. Annealing furnaces for glassware etc. (140,817.) May 11.  
26,561 (1920). Harvey Gas Furnace Co., and Harvey. Furnaces for melting glass. (162,232.) May 4.

## IX.—BUILDING MATERIALS.

## APPLICATIONS.

Briscoe and Faber. Rotary kilns for calcining Portland cement etc. 12,884. May 5.  
Stoederum and Zondervan. Building material. 12,346. Apr. 29.

Tabary. Manufacture of material for buildings, roads, etc. 12,277. Apr. 28.  
Thorne. Heat-conducting powder for covering boilers, pipes, etc., and for forming walls etc. 12,483. May 2.

## COMPLETE SPECIFICATIONS ACCEPTED.

26,168 (1919). Fried. Production of building materials and artificial stones. (134,230.) May 11.  
26,504 (1919). Dyring. Building material. (134,538.) May 11.  
28,509 (1919). Bayer. Manufacture of stone-like materials of foam-like structure. (162,318.) May 11.  
2080 (1920). Jones. Manufacture of disintegrated asbestos for use in making fireproof composition. (162,359.) May 11.  
7389 (1920). Wallace. Treating clayey materials for making bricks, slabs, etc. (162,483.) May 11.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Akt. Ferrolegeringar. Manufacture of manganese alloys poor in carbon, or silicon, or manganese. 12,232. Apr. 28. (Sweden, 12.5.20.)  
Akt. Ferrolegeringar. Production of chromium or its alloys. 12,233. Apr. 28. (Sweden, 12.5.20.)  
Ashcroft. Precipitating precious metals from cyanide solutions. 12,347. Apr. 29.  
Bansen, and Faconneisen-Walzwerk Mannstädt u. Co. Hearth smelting or heating furnaces. 12,793. May 4.  
Blei- u. Silberhütte Braubach A.-G. Treating materials containing precious metals. 12,205. Apr. 28. (Ger., 12.8.20.)  
Burgess. Reduction of aluminium oxide. 12,129. Apr. 27.  
Hibbard. Metallurgical furnaces. 12,391. Apr. 29. (U.S., 1.11.19.)  
Howse. Preserving steel or iron work against corrosion or rusting. 12,507. May 2.  
Jones and Owen. Production of metallic dust. 13,018. May 6.  
Koppers. Desulphurising large masses of iron and steel. 12,244. Apr. 28. (Ger., 28.4.20.)  
Leon and Lister. Dyeing aluminium. 12,413. Apr. 30.  
Patent-Treuhand Ges. Production of drawn tungsten wires. 13,010. May 6. (Ger., 7.5.20.)  
Soc. Anon. Usines Métallurgique de la Basse-Loire. Production of basic steel. 12,260. Apr. 28. (Fr., 18.5.20.)

## COMPLETE SPECIFICATIONS ACCEPTED.

31,791 (1919). Elmore. Treatment of argentiferous lead-zinc sulphide ores. (162,026.) May 4.  
31,936 (1919). Field and others. See VII.  
1240 (1920). Ellis. Production of powdered metals and suboxides. (162,038.) May 4.  
2433 (1920). Maguet. Reducing fused slag to a granular form. (162,375.) May 11.  
2580 (1920). Rare Metals Reduction Co. Production of alloys. (138,348.) May 11.  
2710 (1920). Fletcher. Electrodeposition of metals on iron and iron alloys. (162,391.) May 11.  
5723 (1920). Bradbury, and Rolls-Royce, Ltd. Aluminium alloys. (162,467.) May 11.  
18,964 (1920). Manuf. de Prod. Chim. du Nord Etahl Kuhlmann. Removing dust from the gases derived from the roasting of ores. (147,020.) May 11.  
24,471 (1920). Krupp A.-G. See II.  
6912 (1921). Morgan Crucible Co., and Speirs. See XI.

## XI.—ELECTRO-CHEMISTRY.

## APPLICATIONS.

Chem. Fabr. Griesheim-Elektron, and Suchy. 12,573. See VII.

Hart Accumulator Co., and Holmes. Secondary batteries. 12,703-4. May 3.  
Niblett. Primary and secondary batteries. 11,911. Apr. 26.

COMPLETE SPECIFICATIONS ACCEPTED.

21,585 (1919). Kreher. Electrodes for accumulators. (132,259.) May 11.  
2710 (1920). Fletcher. *See X.*  
16,726 (1920). Metallbank u. Metallurg. Ges., and Lilienfeld. Electrical gas purification (145,477.) May 11.  
6912 (1921). Morgan Crucible Co., and Speirs. Electrically-heated melting and other furnaces. (162,246.) May 4.

XII.—FATS; OILS; WAXES.

APPLICATION.

Hildesheimer and Lion. 12,701. *See XIX.*

COMPLETE SPECIFICATIONS ACCEPTED.

25,932 (1919). Melamid and Grotzinger. Manufacture of products soluble in or forming emulsions with water. (134,223.) May 11.  
2108 (1920). Schwarzkopf. Refining oils and fats. (138,115.) May 11.  
2410 (1920). Bolton. Continuous hydrogenation of unsaturated oils, fats, etc. (162,370.) May 11.  
2544 (1920). Bolton and Lush. Maintaining the activity of metallic catalysts during the hydrogenation of oils, fats, etc. (162,382.) May 11.  
7059 (1920). Stiansen. Refining oils and fats. (141,028.) May 4.  
16,786 (1920). Stiepel. Deodorisation of soaps. (145,502.) May 4.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATION.

Attwater and Crossthaite. Synthetic resin varnish. 12,442. Apr. 30.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Alger and Frood. Fireproofing caoutchouc etc. 12,419. Apr. 30.  
Davidson. Preparation of preservative substances for rubber latex. 12,343. Apr. 29.  
Lambert. Vulcanisation of rubber. 12,132. Apr. 27.

COMPLETE SPECIFICATIONS ACCEPTED.

4288 (1920). Peachy. Vulcanisation of caoutchouc. (162,429.) May 11.  
10,497 (1920). Marshall. Vulcanising processes and apparatus for making highly-expanded vulcanised rubber etc. (162,176.) May 4.  
10,599 (1920). Stevens. Preservation of vulcanised rubber goods. (162,528.) May 11.  
16,438 (1920). Goodyear Tire and Rubber Co. Method and apparatus for vulcanising. (145,425.) May 11.  
2578 (1921). Farrel Foundry and Machine Co. Mixing or masticating rubber etc. (158,279.) May 4.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATION.

Blücher and Krause. Manufacture of plastic material from casein. 12,385. Apr. 29. (Ger., 3.5.20.)

COMPLETE SPECIFICATIONS ACCEPTED.

812 (1917). Bartels. Manufacture of casein specially adapted for producing imitation horn. (162,301.) May 11.

5439 and 5691 (1920). Melamid. Manufacture of artificial tanning substances. (148,268 and 148,738.) May 11.

XVI.—SOILS; FERTILISERS.

APPLICATION.

Boorman and Browning. Treatment of nitrates for fertilisers etc. 12,157. Apr. 27.

COMPLETE SPECIFICATIONS ACCEPTED.

31,584 (1919). Grinnel and Field. Treatment of organic matter for fertiliser purposes. (136,829.) May 4.  
16,034-5 (1920). Badische Anilin- u. Soda-Fabrik. Manufacture of fertilisers. (145,036-7.) May 11.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Chamberlain. Non-poisonous disinfectant. 12,628. May 3.  
Fitzgerald. Preserving fluidity of blood. 12,867. May 5.  
Hildesheimer and Lion. Preparation of a fat for culinary purposes. 12,701. May 3.  
Improved Chilling and Transport, Ltd., and Slann. Antiseptic, disinfectant, or preservative agents, and preserving food products. 12,997. May 6.  
Jaquet. Treatment of grain. 12,535. May 2.  
Plauson's (Parent Co.), Ltd. (Plauson). Dissolving dried or concentrated milk. 12,189. Apr. 28.  
Slann. Preservation of food products. 11,971. Apr. 26.  
Wade (Sykes). Dehydrating apparatus for foods etc. 13,076. May 7.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Barrett Co. 12,153. *See III.*  
Blagden, Nierenstein, and Howards and Sons. Manufacture of amino-derivatives of hydrogenated cinchona alkaloids and their derivatives. 13,004. May 6.  
Rosen. Local anæsthetic. 12,091. Apr. 27.

COMPLETE SPECIFICATIONS ACCEPTED.

17,796 (1920). Boehringer Sohn. Obtaining the active ingredient of *Lobelia inflata*. (145,622.) May 11.  
18,362 (1920). Friederich. Manufacture of tri-nitroresorcin. (162,578.) May 11.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Shawcross. Preparation and treatment of photographic ferric films for lithographic transfer purposes. 12,616. May 3.  
Wedmark. Production of photographic negatives. 12,537. May 2.

XXII.—EXPLOSIVES; MATCHES.

APPLICATION.

Vautin. Separation of nitro-aromatic compounds from explosives etc. 12,458. Apr. 30.

COMPLETE SPECIFICATIONS ACCEPTED.

18,362 (1920). Friederich. *See XX.*  
23,564 (1920). Silberrad. Explosive propellants. (150,002.) May 11.

**L—GENERAL; PLANT; MACHINERY.**

*Corrosion of boiler tubes by carbon dioxide; Unusual* — B. G. Worth. Trans. Amer. Electrochem Soc., 1921, 329—335. [Advance copy.]

SEVERE corrosion of a water-tube boiler using natural water softened by a standard lime-soda process was found to be due to the presence of a soluble bicarbonate of iron,  $\text{H}_2\text{Fe}(\text{CO}_3)_2$ . The bicarbonate decomposed into ferric hydroxide and carbon dioxide on heating the water, and after the introduction of a preheater and the removal of liberated carbon dioxide before entering the boiler, corrosion practically ceased.—C. A. K.

*Rust formation.* E. Sauer. Chem.-Zeit., 1921, 45, 421.

RAPID corrosion of the condensed water outlet pipes of the radiators of a large drying plant was traced to the presence of carbonic acid in the boiler feed-water. When the feed-water was treated by the lime-soda process on the Breda system, thereby reducing the total hardness to about 3 degrees, and eliminating the carbonic acid, the corrosive action of the condensed water practically ceased.—G. F. M.

**PATENTS.**

*Rectifying apparatus; Condensing arrangements of* — E. Barbet et Fils et Cie. E.P. 138,869, 6.2.20. Conv., 8.2.19.

THE cooling coils of the bubbling plates of a rectifying column as described in E.P. 13,539 of 1914 (F.P. 469,979 of 1913; J., 1915, 16) are fed with a portion of the rectified spirit from the combined condenser and cooler, and the rectified spirit thus preheated in the coils is admitted to the top of the column as the reflux fluid. This arrangement prevents formation of scale in the cooling tubes, and may also prevent it in the condenser, for there is no objection to using so large a quantity of condensing (and refrigerating) water that it never reaches the scale-forming temperature. The reflux rectified spirit instead of passing through the worms in the trays may, if desired, be passed through other tubes (or a plate) at the head of the column which will act as a preliminary condenser.—B. M. V.

*Still-head; Spherical* — O. Heublein and E. Weiler. G.P. 328,824, 23.7.19.

THE spherical head is constituted of two spherical jackets, one inside the other, the intervening space being evacuated and the surfaces of the jackets silvered after the manner of a Thermos flask. A tube provided with a cruciform end placed within the spherical head allows of the passage of the vapours to the condenser, such vapours passing through the ends of the cross and through holes in the part of the tube just below the wall of the spherical head. The bottom side of the cross is inclined so that condensed liquid drains back into the distilling vessel.—J. S. G. T.

*Distillation in vacuo; Plant for [continuous] fractional* — E. Lühr. G.P. 332,001, 1.1.20.

THE still can be connected in turn with any one of a number of condensers, each of which may be separately connected with evacuating apparatus and, after evacuation, shut off by a cock. The material to be distilled, e.g., petroleum tar, is supplied from a vessel provided with cocks for shutting off connexion with both the atmosphere and the distillation vessel. The residue after distillation is run off into a vessel which may be evacuated.—J. S. G. T.

*Concentrated solutions [; Centrifugal apparatus for the] manufacture of* — B. Junquera. E.P. 144,240, 9.8.19. Conv., 30.5.19.

THE basket of a centrifugal apparatus has a double wall and double bottom, the cylindrical wall of the inner basket being perforated. The solid to be extracted is placed in the inner basket, and the solvent supplied through an axial opening to the space between the double walls. The centrifugal head of the liquid in the jacket space causes it to flow inwards through the solid matter, after which it may be exhausted over the rim of the outer basket or collected by a scoop and returned to the axial inlet of the same or another apparatus. The solid material may be preheated in a furnace and the extraction apparatus contained in a casing through which the hot gases from the furnace are passed.—B. M. V.

*Centrifugal pulp-thickener.* J. T. Jaeger. U.S.P. 1,374,377, 12.4.21. Appl., 14.7.19.

THE rotating member is tubular and flares to a larger diameter at the discharge end, where there are a non-rotating central outlet for the liquid and a peripheral outlet for the pulp.—B. M. V.

*Grinding, crushing, and dividing granular substances; Apparatus for* — V. Antoine. E.P. 147,686, 8.7.20. Conv., 14.7.17.

IN a disc grinder the driven disc or millstone is mounted on a ball and socket joint on the end of the shaft, the grinding pressure is applied by a weight and lever, and adjustable stops are provided to limit the maximum and minimum grinding space.—B. M. V.

*Mills for grinding and disintegrating materials [; Classifying devices for conical* — H. W. Hardinge. E.P. 150,997, 20.8.20. Conv., 11.9.19.

A conical mill is provided with an additional classifying device consisting of a truncated conical hood surrounding and rotating with the outlet end of the mill which is perforated to allow partly ground material to pass out. The undersize flows away from the classifying hood either over the edge or through perforations. If the mill is mounted on tyres and rollers instead of a journal bearing, the hood may be entirely supplied through the ordinary outlet end of the mill. Alternatively, a baffle may be provided inside the mill proper at about the end of the cylindrical portion, this baffle being perforated with one large central hole and thus acting as a weir, or with many smaller ones and acting as a screen. In all cases a spiral conduit is provided to lead back the oversize from the classification compartment to the main portion of the mill.—B. M. V.

*Grinding mill.* A. E. Jacobson. U.S.P. 1,374,207, 12.4.21. Appl., 7.4.19.

IN a grinding machine with revolving beaters the grinding surface is formed from a segmental plate with loops cut and pressed out of it, the loops remaining attached by their ends.—B. M. V.

*Asbestos cloth, more especially for the dry separation of solid matter from blast furnace gases and the like.* E. Danhardt. E.P. 153,558, 9.7.20. Conv., 31.10.19.

ASBESTOS cloth is made with the warp or the woof composed entirely or partly of threads of loosely spun asbestos; the other threads are of metallic wires or tightly spun asbestos or both. After weaving the cloth may be roughened on both sides.—B. M. V.



*Furnaces for heating crucibles employed for melting or heating metals and/or other materials.* The Crosthwaite Engineering and Furnace Co., Ltd., and J. W. Crosthwaite. E.P. 161,336, 11.2.20.

THE furnace is built up of a number of layers of refractory material which are each pierced with a large central round hole to accommodate the crucible and several other smaller holes (e.g., one at each corner if the exterior shape is square). Each lamina has also fan-shaped recesses formed on each face so that when the laminas are assembled with the outer or corner holes co-axial the pairs of fan-shaped recesses will form fan-shaped ports for the passage of air from the corner ducts to the central crucible chamber. The bottom of the crucible chamber is closed by a refractory hearth, and the whole rests on a hollow bed-plate through which air may be supplied to the vertical corner passages. The tops of the corner passages are closed by plugs so shaped that on rotating through part of a circle a flange will obstruct the upper fan-shaped ports.—B. M. V.

*Fuller's earth; Method of preparing for transit insoluble materials such as —.* L. G. Hill. E.P. 161,419, 8.3.20.

DUSTY material, such as fuller's earth, is mixed with a dilute solution of sodium silicate, and after drying the product is broken into lumps. The material can be recovered in its original finely divided state by treatment with water.—B. M. V.

*Filters.* W. J. Still. E.P. 161,639, 5.1.20.

A BAG filter for large quantities of water carrying small quantities of solid has the framework for the bags constructed of strip metal on three sides and a cast base on the fourth, with coil springs stretched between the base and the further side. The cast base has outlet passages communicating with the interior of the springs, and several units may be clamped together to form a larger unit or block, the whole being enclosed in a pressure-tight casing to which the water is admitted under pressure. Two blocks of filters may be connected with a distributing valve which is automatically operated by rise of pressure (owing to clogging) to switch the liquid over to the other block of filters.—B. M. V.

*Filtering material.* E. Müller. U.S.P. 1,375,532, 19.4.21. Appl., 16.3.17.

THE ashes from vegetable matter, after treatment with a soluble silicate, are washed, dried, and powdered, and a plastic mixture of the product with powdered clay and water is moulded, dried, and baked at a glowing heat.—L. A. C.

*Filter; Automatic —.* A. and A. Sommer. G.P. 330,078, 30.10.18. Conv., 19.2.14.

THE liquor flows upwards through a number of superimposed receivers separated by horizontal partitions. Each receiver is separated by the filter surface into an upper and lower chamber, and the inlet and outlet are situated respectively below and above the filter surface.—L. A. C.

*Drying machine.* T. Allsop and W. W. Sihson, Assrs. to The Philadelphia Drying Machinery Co. U.S.P. 1,374,709, 12.4.21. Appl., 18.12.19.

THE drying chamber contains a conveyor, e.g., a zig-zag continuous chain with carriers for the goods, and a fan, which are driven from the same source of power but can be started and stopped independently; the direction of rotation of the fan may also be reversed.—B. M. V.

*Drying materials; Process and apparatus for —.* E. M. Bassler. U.S.P. 1,374,874, 12.4.21. Appl., 24.3.16. Renewed 16.6.19.

THE material to be dried is carried forward on a suitable conveyor in a closed trunk, and meets a

current of warmed air which is made to pass through the conveyor at intervals by baffle-plates.—B. V. S.

*Drying liquids; Atomising process for —.* Chem. Verwertungs-ges. m.b.H. G.P. 332,000, 10.2.20.

THE liquid is delivered by a pipe to a trough, whence it overflows on to a plate, on the upper side of which it is subjected to a preliminary dispersion by a jet of fluid under pressure directed downwards upon the plate, prior to being atomised into the drying chamber by a similar jet directed upwards from beneath the plate.—J. S. G. T.

*Evaporator.* J. T. Wann, Assr. to R. C. Newell and W. C. Murdoch, jun. U.S.P. 1,375,431, 19.4.21. Appl., 4.5.20.

AN evaporator contains separate storing, heating, and exit chambers, and a drying chamber subdivided into a number of compartments in pairs for receiving the material on trays. Inlet flues on the one side of each of the compartments, and outlet flues on the other side, communicate with the heating and exit chambers respectively, and means are provided for deflecting the heat below each pair of compartments.—L. A. C.

*Evaporation or concentration of solutions, emulsions, and suspensions, and for carrying out chemical reactions; Process and apparatus for —.* G. A. Krausc. G.P. 329,658, 2.5.16.

THE liquid is delivered centrally to an atomising device consisting of a number of tubular arms attached to a chamber rotating with high velocity, the arms being provided with small openings for efflux of the liquid. The atomiser is contained within a chamber through which a current of air is induced, whereby the efficiency of its action is increased.—J. S. G. T.

*Mixing liquids with gases or liquids, or gases with gases; Process and apparatus for —.* A. Wache. G.P. 323,655, 3.8.19.

THE respective fluids are forced under pressure from hollow members through fine jets tangentially into a mixing chamber, the respective discharges being in parallel planes one above the other and in counter directions. The fluid discharged through the lower system of jets ascends spirally in the mixing chamber and meets the oppositely directed stream of the other fluid, discharged from the upper system of jets, at the narrowest section of the mixing chamber, which tapers gradually.—J. S. G. T.

*Filling material having the form of a truncated cone for reaction columns etc.; Annular —.* Stollawerk A.-G. G.P. 324,442, 14.11.17.

THE filling material is built up of rings in such manner that the inner and outer surfaces of the resulting structure have the form of truncated cones tapering in opposite directions. This form of packing takes up only about 35% of the space of the reaction chamber, compared with 40–60% taken up by other kinds of filling material.—J. S. G. T.

*Saturated solutions; Apparatus for producing —.* W. Otte. G.P. 323,218, 8.11.19.

THE substance to be dissolved is supported in a series of superposed rings of inverted frusto-conical shape with their axes vertical, the diameter of successive rings increasing in the downward direction. The floor of the lowest ring upon which the substance rests is perforated for the passage of solvent. The device rests upon a down pipe, and is contained within a vessel holding the solvent, the top cone being above the level of the liquid.—J. S. G. T.

*Heating or cooling device with scrapers.* F. Fiedler, Dampfkesselfabrik, Maschinen- u. Apparate-Bauanstalt, O. Kittel, and F. Hornung. G.P. 328,761, 26.4.19.

A SERIES of cylindrical or hemi-cylindrical heating or cooling chambers is disposed with their axes parallel and adjacent to one another, and scrapers are arranged so as to remove any matter deposited upon the cylindrical surfaces, each scraper being effective over a half cylinder. A conveyor or similar device traversing the length of the apparatus and disposed above a channel serves for the removal and collection of material removed by the scrapers.

—J. S. G. T.

*Steam boilers; Method of maintaining low pressure — free from mud and scale.* Maschinenbau-A.-G. Balcke. G.P. 331,279, 27.6.18.

SUFFICIENT hydrochloric acid is added to the feed water to convert carbonates into soluble chlorides; these salts are not decomposed at the comparatively low temperature, so that the water can be evaporated to 15% concentration without formation of scale or damage to the shell from free acid.—C. I.

*Gases; Method of separating and recovering — and apparatus therefor.* W. A. Patrick, B. F. Lovelace, and E. B. Miller. E.P. 137,284, 24.12.19. Conv., 28.12.18.

SEE U.S.P. 1,335,348 of 1920; J., 1920, 392 A.

*Filtering apparatus; Rotating-screen —.* The Dorr Co., Assees. of C. L. Peck. E.P. 139,493, 25.2.20. Conv., 5.10.18.

SEE U.S.P. 1,338,999 of 1920; J., 1920, 437 A.

*Pulveriser.* H. T. Rudisill, Assee. of J. H. and J. Macartney. E.P. 148,369, 9.7.20. Conv., 10.1.17.

SEE Reissue of U.S.P. 1,253,619 of 1918; J., 1920, 52 A.

*Ball-mill for crushing ores.* M. Vogel-Jorgensen, Assr. to F. L. Smidth & Co. U.S.P. 1,374,410, 12.4.21. Appl., 20.8.19.

SEE E.P. 125,064 of 1919; J., 1919, 886 A.

*Crushing-mill of the roller and ring type.* C. M. Conder and G. T. Vivian. U.S.P. 1,374,823, 12.4.21. Appl., 10.12.20.

SEE E.P. 159,244 of 1919; J., 1921, 248 A.

*Gases; Art of [electrically] separating suspended particles from —.* E. Müller, Assr. to The Chemical Foundation, Inc. U.S.P. 1,357,466, 2.11.20. Appl., 11.8.11.

SEE Addition to F.P. 449,337 of 1912; J., 1914, 601.

*Crushing apparatus; Gyratory —.* J. E. Kennedy. E.P. 139,216, 20.2.20. Conv., 23.10.17.

*Supplying granular or pulverulent substances to furnaces or the like; Apparatus for —.* D. Wright. E.P. 161,250, 2.1.20.

*Furnaces; Forced draught — [for pulverulent fuel].* W. P. Thompson. From H. Cruse und Co. E.P. 161,488, 27.7.20.

*Condenser.* U.S.P. 1,374,357. See IIa.

*Furnaces.* E.P. 147,190. See X.

*Centrifugal apparatus.* E.P. 161,822. See XVII.

## IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

*Tar fog, dust, and water in producer gas; Determination of —.* E. Jenkner. Stahl u. Eisen, 1921, 41, 181—183.

It is essential to cool hot gas before filtering to separate suspended matter, otherwise a fractionation of any deposited tar may occur. The gas is drawn at the rate of 60 l. per hr. through a glass tube 30 cm. long, one portion of which 10 cm. long and 2 cm. diam., with a nozzle of 6 mm. diam., is water-cooled, and projects into the gas main. The after portion of the tube, 20 cm. long and 4 cm. diam., is packed with Raschig rings of glass, 5 mm. diam. These retain all the dust and practically all the tar. The residue is retained by a weighed glass tube packed with cotton wool. This is followed by two weighed U-tubes containing calcium chloride to retain water. To separate the tar and dust in the first tube, the tar is extracted with warm benzene, and the weight of dust retained by the Raschig rings is obtained. The benzene solution is filtered through a tared filter to obtain the weight of dust lost in the washing. The weight of tar can then be deduced.—H. J. H.

*Petroleum residues; Method of determining the density of —.* C. Predescu. Bul. Sci. Acad. Roumaine, 1920, 6, 148—150.

A SP. GR. bottle is weighed empty ( $p_1$ ), a small piece of the petroleum residue is then pressed on the interior wall of the bottle and the latter again weighed ( $p_2$ ); the bottle is then filled with water at 15° C. and again weighed ( $p_3$ ). If  $p_2$  is the weight of the bottle filled with water alone, then the sp. gr. ( $x$ ) of the petroleum residue is given by the formula  $x = (p_3 - p_1) / \{ p_2 + p_3 - (p_1 + p_2) \}$ .—W. P. S.

*Petroleum; Refractive index of Roumanian —.* C. Predescu. Bul. Sci. Acad. Roumaine, 1920, 6, 150—156.

THE refractive index of Roumanian petroleum increases with the boiling point of the fractions, the increase being greater than is the case with the specific gravity. The fraction, h.p. 50°—60° C., has  $n_D^{20} = 1.384$  and sp. gr. 0.6830, whilst the fraction, h.p. 340°—350° C., has  $n_D^{20} = 1.526$  and sp. gr. 0.9051.—W. P. S.

*Petroleum; Optical rotation of Roumanian —.* C. Predescu. Bul. Sci. Acad. Roumaine, 1920, 6, 181—188.

CERTAIN Roumanian petroleums, particularly the higher boiling fractions of these oils, contain optically active substances. As the colour of the fractions interferes with observation in the polariscope, even when the material is diluted with a solvent, the petroleum was filtered through fuller's earth under pressure before examination. Two oils examined in this way had  $[\alpha]_D^{20} = +1.19$  and  $+1.52$  respectively.—W. P. S.

*Petroleum; Capillary constants of Roumanian —.* C. Predescu. Bul. Sci. Acad. Roumaine, 1920, 6, 188—196.

THE capillary constant  $a$ , or the surface tension, is found from the formula  $a = d \times r(h + \frac{1}{2}r)/2$ , where  $r$  is the radius of the capillary,  $d$  the sp. gr. of the petroleum, and  $h$  the height of the capillary column of oil. The value increases with the h.p. of the petroleum fraction; for the fraction, h.p. 50°—60° C.,  $a = 1.92$ , and for the fraction, h.p. 340°—350° C.,  $a = 3.18$ . The viscosity of the fractions increases more rapidly than does the capillary constant.

—W. P. S.

*Light petroleum (pentane, hexane, heptane, octane); Narcotic action of —.* H. Fühner. *Biochem. Zeits.*, 1921, 115, 235–261.

In contradistinction to ether and to chloroform, the hydrocarbons mentioned when inhaled (by mice and rats) often cause great excitement, and soon affect the respiration adversely; benzene is an even more powerful excitant. In molecular quantities benzene is a somewhat less powerful anæsthetic than chloroform; ether has only one-quarter of its activity. Hexane and ether, heptane and benzene, octane and chloroform have about the same degree of activity. The activity in the homologous series, pentane, hexane, heptane, octane, increases approximately in the ratio 1:3:3'..... The solubility in water diminishes in the same ratio.—G. B.

*Blast-furnace gas.* Fowles. See X.

#### PATENTS.

*Coking retort-oven.* J. Becker, Assr. to The Koppers Co. U.S.P. 1,374,546, 12.4.21. Appl., 8.11.20.

THE coking chambers have contiguous heating walls with vertical combustion flues. Tapered horizontal flues in each heating wall connect a number of the combustion flues of their respective heating walls, the horizontal flues also communicating with the corresponding horizontal flues of an adjacent heating wall. Reversible regenerators, which can be regulated individually, extend crosswise parallel with the coking chamber and the heating walls, and are connected in pairs with combustion flues of a single heating wall. Means are provided for controlling the individual flow through individual regenerators and their connected groups of combustion flues.—A. G.

*Constituents of the distillation gas from fuel (e.g., ethylene); Process for the separate recovery of —.* F. Bergius and P. Kalnin. E.P. 146,332, 2.7.20. Conv., 26.10.17.

THE gas is freed from tar, ammonia, and benzene, and is then passed over adsorbent charcoal at 0° to -20° C. The adsorbed gas is then expelled and again subjected to the same treatment. By repeated adsorption the ethylene content can be greatly increased, e.g., from 1–2% successively to 17, 36, and 59%.—A. G.

*Purifying coal gas; Method of — by means of ammonia.* C. Still. E.P. 147,583, 8.7.20. Conv., 10.12.17.

THE scrubber first traversed by the cooled crude gas is sprayed with the highly concentrated final product of the ammonia-recovery plant, and the subsequent scrubber is sprayed with the weaker ammonia solution from the reflux condenser of this plant. The gas flows in series through further scrubbers which are traversed in counter current, first by condensed liquor from the crude gas which contains little ammonia, and then by fresh water for the purpose of completely or partially washing out the ammonia. The residue of ammonia remaining in the gas after passing the ammonia scrubbers is recovered, together with the ammoniacal vapours arising from the ammonia recovery plant, and containing hydrogen sulphide and carbon dioxide, in a saturating plant in the form of ammonium sulphate, for which the necessary sulphuric acid is produced from the hydrogen sulphide remaining in the exhaust gases from the saturator. When the whole of the ammonia is removed in the scrubbing plant the gas that leaves the recovery plant is washed with the ammoniacal liquor condensed in the preliminary cooling of the original crude gas, so as to return the ammonia carried by the first-named gas to the ammonia-recovery plant.—A. G.

*Soot-carbon, retort-graphite and other carbon products; Process for the production of — from natural gas.* Rütgerswerke A.-G. E.P. 137,065, 24.12.19. Conv., 12.6.18.

NATURAL gas is decomposed to the extent of 65% only in a retort, and the exit gases are used for firing the retort. The degree of decomposition is regulated by varying the speed of the gas, which may be preheated by the exhaust from the combustion chamber.—C. I.

*Petroleum jelly; Process for making a substitute for —.* Rütgerswerke A.-G. E.P. 134,528, 1.10.19. Conv., 26.10.18.

POWDERED pitch of m.p. about 50°–60° C., containing 50% or more of aliphatic hydrocarbons, produced, e.g., by distilling low-temperature coal-tar, or lignite tar, at about 300° C., under reduced pressure, is treated below its softening point with petroleum ether and/or benzene, and/or other solvents, in sufficient quantity to yield a homogeneous, viscous mixture. The solvent is expelled after separation of the insoluble matter, yielding a product which, after purification and decolorisation by the usual means, can be employed as a substitute for petroleum jelly.—L. A. C.

*Petroleum and analogous distillates; Process of cleaning and refining —.* A. J. Paris, jun. E.P. 161,253, 2.1.20.

THE distillates are compressed in the form of spray, vapour, or mist, in the presence of a permanent gas free from oxygen and a neutral purifying liquid, such as glycerin, Turkey-red oil, mineral lubricating oil, or castor oil, under such conditions of temperature and pressure (which may range from 25 to 350 lb. per sq. in.) that cracking of the distillates is avoided. A mixture of the gas with oil vapour, prepared by treating the distillates or crude oil with the gas at a suitable temperature, may be fed into the compression chamber, or the gas and oil may be fed in independently. After the treatment the gas and purifying liquid are separated from the oil vapour, which is subsequently condensed, and are returned to the apparatus for treating more oil.—L. A. C.

*Crude oil; Process of recovering —.* I. S. Joseph. U.S.P. 1,362,105, 14.12.20. Appl., 29.4.19.

To recover crude oil from emulsions, such as "cut oil," "bottom settlings," etc., the emulsion is subjected to centrifugal action so as to cause the constituents of different specific gravity to move along different paths, and a liquid of approximately the same specific gravity as the heavier constituent is introduced into the separator and caused to move along the path of the heavier constituent with a velocity greater than that of the latter, so as to carry along solid impurities which would otherwise be deposited.

*[Air condenser for use in] apparatus for treating hydrocarbons.* J. W. Coast, jun., Assr. to The Process Co. U.S.P. 1,374,357, 12.4.21. Appl., 31.5.17.

A HORIZONTAL cylindrical vessel, fitted internally with horizontal tubes open at both ends, is separated into a number of chambers by baffles open alternately at the top and bottom to provide a sinuous path for the hydrocarbon vapours or the like which are led into the bottom of the vessel at a point near one end. A current of air, induced by connecting all the tubes, at the end near the inlet for hydrocarbon vapours, with a common flue, passes through the tubes and cools the vapours to progressively lower temperatures as they pass through the vessel, causing condensation of different fractions in the separate chambers, whence they are withdrawn.—L. A. C.

*Petroleum-oils; Apparatus for distilling* — A. D. Smith. U.S.P. 1,374,402, 12.4.21. Appl., 13.3.17.

A STILL consists of a vessel with a sloping bottom and a number of sloping tubes below the vessel connected at their ends with the bottom of the vessel, which is also connected with a well. The circulation of oil induced by heating the tubes and the vessel diminishes the deposition of carbon in the apparatus.—L. A. C.

*Hydrocarbon oils; Process of converting higher-boiling-point — into lower-boiling-point hydrocarbon oils.* W. M. McComb. U.S.P. 1,374,858, 12.4.21. Appl., 3.7.20.

A MIXTURE of the oil with a smaller quantity of steam is passed through a coil heated at the inlet and outlet ends to the initial and final b.p. of the oil respectively, and at intermediate points to progressively higher temperatures increasing at the same rate as the b.p. temperatures of the oil increase in fractional distillation of a sample of the oil.—L. A. C.

*Hydrocarbons; Process of distilling* — W. C. Averill, jun. U.S.P. 1,375,245, 19.4.21. Appl., 12.7.19.

IN distilling crude petroleum oil containing material quantities of asphalt-forming material and having a high sulphur content, e.g., Gulf Coast crude petroleum, to produce pale lubricating oil and distillates, including gasoline, a portion of the products of combustion employed for heating the still bottom is withdrawn from the flue, cooled, and passed through the oil in the still. The hydrocarbon vapours are subsequently condensed from the mixture of gas and vapour issuing from the still.

—L. A. C.

[*Mineral*] *oil; Apparatus for refining* — S. F. Stephens, Assr. to W. J. Boyle, sen., and C. G. Grant. U.S.P. 1,375,427, 19.4.21. Appl., 14.11.18. Renewed 24.1.21.

THE oil is fed on to a rotating, horizontal plate within a still and flows as a thin film towards the periphery of the plate, whence it is discharged as a fine spray on to the walls of the still.—L. A. C.

*Mineral oil hydrocarbons; Conversion of — into low- and high-boiling products.* Deutsche Erdöl A.-G., F. Seidenschur, and C. Koettwitz. G.P. 302,585, 29.1.16.

THE oil is treated by one of the known cracking processes, and the benzene produced is removed from the products, which are then distilled to obtain a product having a boiling point and specific gravity near to that of the original oil. This is then put through the cracking process again, either alone or mixed with a proportion of the original oil. The process gives a larger yield of benzene which is free from the usual objectionable odour of benzene obtained by a cracking process, while the residue from the distillation is a more valuable lubricant than the usual tarry residue that is obtained, and a smaller quantity of gas is produced. The total loss in the process is about 8%.—A. R. P.

*Lubricating-oil.* R. H. Brownlee. U.S.P. 1,374,277, 12.4.21. Appl., 23.11.18.

THE oil produced as described in U.S.P. 1,309,432 (J., 1919, 621 A) has a flash point of 425°–450° F. (218°–232° C.), and a cold test of approximately –4° F. (–20° C.) for a cut having a viscosity of 76 at 212° F. (100° C.).—L. A. C.

*Viscous [lubricating] oils; Manufacture of* — Tetralin G.m.b.H. G.P. 333,060, 28.7.18. Addn. to 319,799 (J., 1920, 623 A).

FORMALDEHYDE, polymerisation products of formaldehyde, or substances capable of yielding formal-

dehyde, are allowed to react with hydronaphthalenes, with or without addition of condensing agents, but in absence of acids. By heating tetrahydronaphthalene with paraformaldehyde and phosphorus pentoxide a highly viscous oil (probably ditetrahydronaphthylmethane), b.p. 257°–258° C. at 15 mm., is formed, and also a brittle, light-coloured resin. If acids are used as condensing agents more resin is formed and the yield of oil is reduced. The viscous products may be used as lubricants, especially for refrigerating machines and aircraft engines. As the oils are easily sulphonated their sulphonic acid salts can be made use of for the manufacture of drilling oils and the like.—C. A. C.

*Fuel; Liquid* — U.S. Industrial Alcohol Co., Assecs. of A. A. Backhaus. E.P. 133,709 10.10.19. Conv., 28.11.17.

SEE U.S.P. 1,324,765 of 1919; J., 1920, 99 A.

*Fuels; Liquid* — U.S. Industrial Alcohol Co., Assecs. of A. A. Backhaus. E.P. 140,797, 25.3.20. Conv., 12.10.17.

SEE U.S.P. 1,271,115 of 1918; J., 1918, 539 A.

*Peat; Process of drying raw* — Nasspress-Ges. E.P. 146,263, 28.6.20. Conv., 2.1.14.

SEE U.S.P. 1,143,497 of 1915; J., 1915, 1003.

*Lignites; Carbonising of* — International Coal Products Corp., Assecs. of W. Runge. E.P. 142,443, 17.1.20. Conv., 30.4.19.

SEE U.S.P. 1,334,170 of 1920; J., 1920, 325 A.

*Gas; Method of producing* — O. U. Bean. E.P. 162,159, 19.3.20.

SEE U.S.P. 1,337,637 of 1920; J., 1920, 439 A.

*Coke discharging apparatus for coke oven batteries.* L. Wilputte. E.P. 161,904, 22.9.20.

*Gas generators; Blast heaters and gas coolers for* — Eisenwerk Jagstfeld Ges. E.P. 145,540, 22.6.20. Conv., 17.5.18.

*Distillation.* G.P. 332,001. See I.

*Ammonia etc.* G.P. 328,829. See VII.

*Fatty acids etc.* G.P. 332,594. See XX.

*Gas analysis apparatus.* E.P. 138,355, 160,854, and 160,930. See XXIII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Charcoal; Production of artificially dense* — [for gas-masks]. L. F. Hawley. J. Ind. Eng. Chem., 1921, 13, 301–302.

As a substitute for coconut shell for the manufacture of charcoal for gas-masks, the residue, obtained by hydrolysing sawdust with dilute acid and then leaching out the sugar, has proved to be more satisfactory as regards yield, density, and activity of the charcoal than any untreated wood. For small-scale experiments, 2-in. briquettes compressed at 35,000 lb. per sq. in. from 20-mesh sawdust, to a density of 1.21, were distilled in a 2.5-in. pipe, enclosed in a 4-in. pipe, pressure being maintained during the operation by means of a screw and falling weight. At about 300 lb. per sq. in. and a maximum temperature of 450° C., a 40% yield was obtained, the charcoal having an apparent density of 0.62 and a chloropicrin absorption value of 700 mins., as compared with 0.63 and 900, respectively, for coconut shell charcoal. The product was hard and shiny, resembled anthracite.

coal, and had a conchoidal fracture. On a semi-commercial scale with a retort 10 ft. in length, the best results were obtained by slowly distilling 4-in. briquettes, made from 4- to 43-mesh dust, under pressures varying from 80 to 130 lb. per sq. in.; the product had an apparent density of 0.58 and a chloropicrin absorption value of 600. The less satisfactory results obtained on the large scale may be due to the vapours not escaping readily from the larger briquettes and to the coarser raw material used.—W. J. W.

#### PATENTS.

*Retorts for the distillation of carbonaceous substances.* Low Temperature Carbonisation, Ltd., M. Davidson, and H. L. Armstrong. E.P. 161,608, 14.10.19.

COAL or other carbonaceous substance is distilled at low temperature in relatively thin layers under such conditions that the gas and vapour evolved throughout the charge in the retort are rapidly drawn away from the hot retort walls, to which the heat is applied, into a central space or chamber, whence they are immediately withdrawn. This central space is capable of contraction to accommodate the expansion of the charge during distillation. In the case of vertical retorts, a collapsible casing or equivalent is used, and means are provided for permitting the inward movement of the plates constituting the collapsible casing. Steam may be admitted to the retort.—A. G.

*Wood distillation.* E. M. Sawtelle, Asst. to J. P. Carter and R. L. Squibb. U.S.P. 1,374,887, 12.4.21. Appl., 10.9.18.

SUBDIVIDED wood is fed continuously into a producer, air being blown in at the bottom, but no steam being injected. The products of distillation are led off into a condensation system from which the pyrolytic liquids are removed.—A. G.

*Impregnated wood; Recovery of the preserving material from —.* H. Wiedemann. G.P. 334,307, 16.11.19.

IMPREGNATED wood is subjected to fractional distillation whereby, in addition to the usual products of wood distillation, the substances, such as anthracene or creosote oils, or copper sulphate, which were used to preserve the wood, are also recovered. The distillation may be carried out in the usual way or, after addition of water, under increased pressure.—A. R. P.

*Liquids of high organic content; Process of destructively decomposing — and product of such process.* A. H. White. U.S.P. 1,374,889, 12.4.21. Appl., 24.7.19.

AN aqueous liquid, containing a large amount of organic matter and capable of yielding valuable carbon compounds upon destructive distillation, is intimately mixed, in a concentrated condition, with sufficient quicklime to heat the mass autogenously to a temperature at which destructive distillation occurs, and the volatile compounds are distilled off, at least the major portion of the heat required being applied by the heat of the reaction. The residue is calcined to produce a fresh charge of lime.—A. G.

*Electrodes for electric searchlights.* Optische Anstalt C. P. Goerz A.-G. E.P. 148,450, 10.7.20. Conv., 24.9.18.

THE formation of soot on electrodes used in searchlights which are operated on current overload is reduced by the addition of oxides or silicates, such as cerium oxide, silica, etc., to the electrodes. Alternatively, the electrodes may be surrounded with a casing of silicates.—J. S. G. T.

### III.—TAR AND TAR PRODUCTS.

*Phenoxides; Decomposition of — by heat.* F. Fischer and U. Ehrhardt. Ges. Abhandl. Kennt. Kohle, 1919, 4, 237–263. Chem. Zentr., 1921, 92, I., 762–763.

INVESTIGATION of the products obtained by the dry distillation of a number of normal and basic metal phenoxides and cresyl-oxides showed that sodium and potassium phenoxides yield solely gaseous products, chiefly hydrogen and a little methane, whereas the compounds with other metals yield varying proportions of liquid products in addition to the above and other gases.—L. A. C.

*Naphthalene; Exhaustive sulphonation of —.* H. E. Fierz and F. Schmid. Helv. Chim. Acta, 1921, 4, 381–387.

ACCORDING to the results of Armstrong and Wynne (1885–1895), who showed that sulpho-groups never enter the naphthalene nucleus in the ortho-, para-, or peri-position to one another, exhaustive sulphonation of naphthalene can lead to only two products, namely, the 1.3.6-trisulphonic and 1.3.5.7-tetrasulphonic acids. The authors are, indeed, unable to confirm the statement (G.P. 79,054) that these two acids are accompanied by Senhofer's naphthalenetetrasulphonic acid (Monatsh., 1882, 3, 112) when naphthalene is heated at 260° with concentrated sulphuric acid and phosphorus pentoxide. The apparent losses attending the manufacture of H-acid are dependent on the formation of the tetrasulphonic acid and on oxidative destruction of naphthalene. Together with the tetrasulphonic acid, the 2.7- and the 1.6-disulphonic acids are often formed in small proportions, these acids evidently undergoing further sulphonation to the 1.3.6-trisulphonic acid only with difficulty. 1-Nitronaphthalene-3.6.8-trisulphonic acid is described. (Cf. J.C.S., June.)—T. H. P.

*Phenols of low-temperature tar.* Gluud and Breuer. See XIII.

*Lead compounds of phenols.* Fischer and Ehrhardt. See XIII.

*Benzoic acid from benzene.* McKee and Strauss. See XX.

#### PATENTS.

*Tar-oils, especially from lignite; Process for refining — with aqueous alkalis.* Deutsche Erdöl A.-G. G.P. 333,061, 24.10.18.

THE oil is intimately mixed with the alkali solution by passing a current of steam through the two liquids. The mixture is then distilled with steam at temperatures up to 300° C. and eventually under reduced pressure. No separation of tarry and asphalt-like components takes place, and the distillate obtained is very pure and practically odourless.—A. R. P.

*Pyridine bases; Manufacture of —.* Farb. vorm. Meister, Lucius, und Brüning. E.P. 147,101, 7.7.20. Conv., 23.12.18. Addn. to 146,869 (J., 1921, 54).

FORMALDEHYDE or acetone is substituted for a part of the paraldehyde. Thus, when 15 pts. of formaldehyde (30%) is heated with 8.7 pts. of ammonia (25%) and 13.2 pts. of paraldehyde a mixture of bases is obtained consisting of about 50% of aldehyde-collidine and 50% of methylpyridines, chiefly  $\alpha$ -picoline. Ammonia, paraldehyde, and acetone give a product consisting almost entirely of trimethylpyridine.—G. F. M.

**1.4-Naphthylenediamine and its sulphonic acids; Manufacture of arylsulphonyl and arylenedisulphonyl derivatives of —.** G. T. Morgan, and The Imperial Trust for the Encouragement of Scientific and Industrial Research. E.P. 160,853, 6.8.19.

**ARYLSULPHONYL-1.4-NAPHTHYLENEDIAMINES**, and arylenedisulphonyl-bis-1.4-naphthylenediamines, and the sulphonic acids of these compounds can be prepared by the reduction in a not too strongly acid or alkaline reducing medium of the *p*-azo derivatives of the corresponding arylsulphonyl- or arylenedisulphonyl-naphthylamines or their sulphonic acids containing the sulphonic group in the 2, 6, 7, or 8 positions. The reduction can be carried out with such reagents as zinc dust and sodium hydroxide, zinc dust and ammonium chloride, iron borings and dilute acids, alkaline sodium hydro-sulphite, stannous or titanous chloride, etc., provided the medium is not sufficiently acid or alkaline to effect hydrolysis of the arylsulphonyl groups. Thus, for example, toluene-*p*-sulphonyl-1.4-naphthylenediamine is obtained by condensing  $\alpha$ -naphthylamine with *p*-toluenesulphonic chloride in presence of sodium acetate, dissolving the product in sodium hydroxide, and treating with benzene-diazonium chloride, whereby sodium benzene-4-azo-toluene-*p*-sulphonyl- $\alpha$ -naphthylamine separates; this is converted into the free azo compound, and the latter reduced with zinc and ammonium chloride in aqueous alcoholic solution. (Cf. J.C.S., June.)  
—G. F. M.

**Fluorene; Catalytic oxidation of —. Process of producing maleic acid. Process of producing anthraquinone. Catalytic oxidation of naphthalene.** (A) J. M. Weiss and C. R. Downs, (b, c, and d) C. R. Downs, Assrs. to The Barrett Co. U.S.P. 1,374,695, and 1,374,720-2, 12.4.21. Appl., (A) 21.11.19, (b and c) 3.7.20, and (d) 10.7.20.

(A) **FLUORENE** is oxidised to fluorenone by treatment at 300°–700° C. with a gas containing oxygen in the presence of a metallic oxide as catalyst. The use of mercury out of contact with the reaction mixture but in heat-transferring relationship thereto is claimed in the manufacture of (a) maleic acid and (c) anthraquinone by the partial oxidation of benzene and anthracene respectively by a gas containing oxygen in the presence of a catalyst. (b) Naphthalene vapour is partially oxidised by treatment with a gas containing oxygen in the presence of aluminium oxide as catalyst.—L. A. C.

**$\beta$ -Nitronaphthalene; Manufacture of —.** Tetralin G.m.b.H. G.P. 332,593, 7.10.19.

A **MIXTURE** of mononitrotetrahydronaphthalenes obtained according to G.P. 299,014 (J., 1920, 174 A) is subjected to fractional distillation;  $\beta$ -nitrotetrahydronaphthalene is separated from the first fractions by freezing and is dibrominated and the product distilled. By a vacuum distillation of the thick, oily, brominated product hydrobromic acid is split off at about 150° C. and  $\beta$ -nitronaphthalene, m.p. 76° C., distils over at 190°–200° C. at 20 mm.  $\beta$ -Nitronaphthalene cannot be obtained by direct nitration of naphthalene.—C. A. C.

**1.4-Chloronitroanthraquinone; Manufacture of —.** F. Ullmann. G.P. 332,853, 17.8.16.

**2.4-DINITRO-1-HYDROXYANTHRAQUINONE** is treated with arylsulphochlorides in presence of substances capable of combining with acid. If the reaction is allowed to take place in presence of potash the hydroxyl group is replaced by chlorine and 1-chloro-2.4-dinitroanthraquinone is formed. If diethylaniline is used a good yield of 1.2-dichloro-4-nitroanthraquinone is obtained. 2.4-Dinitro-1-hydroxy-

anthraquinone, prepared from 1-hydroxyanthraquinone and nitric acid (sp. gr. 1.52) in sulphuric acid solution, forms yellow needles (from glacial acetic acid), m.p. 248° C.; it is slightly soluble in hot alcohol and hot glacial acetic acid, easily soluble in nitrobenzene. 1-Chloro-2.4-dinitroanthraquinone crystallises from glacial acetic acid in slightly yellow needles, m.p. 240° C., slightly soluble in hot alcohol, soluble in benzene, easily soluble in nitrobenzene; by heating with  $\beta$ -naphthylamine in amyl alcohol solution the purple compound 2.4-dinitro-1- $\beta$ -naphthylaminoanthraquinone is obtained. 2.4-Dinitro-1-hydroxyanthraquinone heated with *p*-toluenesulphochloride and diethylaniline gives 1.2-dichloro-4-nitroanthraquinone, yellow needles from benzene, m.p. 246° C., slightly soluble in alcohol, soluble in benzene, easily soluble in nitrobenzene.—C. A. C.

**Liquors containing phenoloid bodies; Purification of —.** The Koppers Co., Asscs. of C. A. Basore. E.P. 139,168, 14.2.20. Conv., 15.2.19.

SEE U.S.P. 1,323,239 of 1919; J., 1920, 80 A.

**Tar; Method of treating hydrocarbons derived from gas —.** M. Melamid, Assr. to The Chemical Foundation, Inc. U.S.P. 1,362,127, 14.12.20. Appl., 4.8.14.

SEE G.P. 278,192 of 1913; J., 1915, 269.

See also pages (A) 375, *Distillation* (G.P. 332,001). 378, *Petroleum jelly substitute* (E.P. 134,528). 379, *Viscous oils* (G.P. 333,060). 399, *Communiting pitch* (G.P. 333,704). 412, *Fatty acids etc.* (G.P. 332,594).

#### IV.—COLOURING MATTERS AND DYES.

**Anthocyanins and anthocyanidins. IV. (a) Anthocyan colours in flowers. (b) Formation of anthocyanins in plants.** A. E. Everest and A. J. Hall. Proc. Roy. Soc., 1921, 92 B, 150–162.

For the most part a reply to Shibata and others (J., 1919, 217 A) and a criticism of their work. Further experiments are described in support of the view that in nature yellow sap pigments of the flavonol group are first formed and then the anthocyan colouring matters are produced from them by reduction.—W. G.

#### PATENTS.

**Disazo-dyes for wool; Preparation of —.** Badische Anilin- und Soda-Fabr. G.P. 333,077, 3.5.19. Addn. to 330,824 (J., 1921, 294 A).

THE tetrazo-compound of 4.4'-diaminobenzophenone is combined with 2 mols. of 2.6- or 2.7-naphthol-sulphonic acid. The resulting dyes give various shades of scarlet on wool.—A. R. P.

**Colour of the anthraquinone series; Manufacture of a new —.** L. Cassella und Co. E.P. 148,339, 9.7.20. Conv., 15.3.15.

SEE U.S.P. 1,285,727 of 1918; J., 1919, 130 A.

**Catechin.** E.P. 161,431. See XV.

**Photosensitising dyes.** U.S.P. 1,374,871-2. See XXI.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Wool; Harmful action of acids on —.** M. Becke. Textilber., 1921, 2, 194–195.

WHEN treated with acids wool acquires an acid character so that its resistance to treatment with alkalis is diminished. When samples of wool were steeped for 2 hrs. in solutions containing respectively hydrochloric, sulphuric, oxalic, formic, and



acetic acids, 0.9, 0.4, 0.4, 0.225, and 0.1% of the wool dissolved. The same samples were washed, dried overnight, neutralised with a very dilute solution of sodium bicarbonate, and boiled for 2 hrs. in a 4% (on weight of wool) solution of Marseilles soap, whereby a further 1.8, 2.0, 1.35, 1.15, and 1.0% of the wool dissolved. When different samples of wool were immersed for 2 hrs. in solutions containing 0, 2, 4, 6, 8, and 10% respectively of sulphuric acid, whereby 0.35, 0.20, 0.45, 0.75, 1.10, and 1.50% of the wool dissolved, and were then washed, neutralised in a dilute solution of sodium bicarbonate, and heated for 2 hrs. at 95°–100° C. in a solution containing 2.5% of sodium carbonate, a further 1.2, 1.8, 3.0, 4.4, 5.2, and 7.6% of the wool dissolved. When samples of wool, after immersion in solutions containing 4% of sulphuric acid and 0, 10, 25, 50, 100, and 200% of Glauber's salt, whereby 0.45, 0.375, 0.35, 0.30, 0.275, and 0.20% of the wool dissolved, were treated with a 4% solution of soap at 95°–100° C., a further 2.0–1.9% of the wool dissolved. Under the same conditions, except that acetic acid (5% instead of 4%) and sodium acetate were used in place of sulphuric acid and Glauber salt, 0.10, 0.125, 0.20, 0.24, 0.25, and 0.25% of the wool dissolved, and in the subsequent soaping the amount of wool which dissolved was 1.20–1.30%.—A. J. H.

*Retting; Investigations of the process of* —. E. Kayser and H. Delaval. Bull. Soc. d'Encour., 1920, 132, 277–296.

The conditions affecting the retting of fibrous materials such as flax, hemp, ramie, jute, and nettles by means of six different kinds of micro-organisms isolated from macerations of flax, hemp, jute, etc., have been investigated; all of the micro-organisms were aerobic except one, which was a facultative anaerobe. Sterilisation of the materials before retting was effected by means of alkali hypochlorite solution or preferably with carbon bisulphide vapour. The optimum temperature for four of the organisms used was 25°–30° C., and for the others 32°–35° C. Manganese salts hindered retting, and aeration of the retting liquors was beneficial in one instance and detrimental in another. In one experiment the gas liberated in the early stages of retting contained 65% of carbon dioxide and 35% of nitrogen, and in the later stages it contained 75% and 25% respectively, while the retting liquor contained alcohol and a much smaller amount of acetic acid. By the selection of suitable bacilli and conditions the retting process may be scientifically controlled so as to give uniform results.—A. J. H.

*Reeds (Phragmites communis, Trin.); Composition of* —. F. Herig. Cellulosechem., 1921, 2, 25–34. (Cf. Heuser, J., 1920, 744 a.)

In an investigation of the composition of reeds (*Phragmites communis*), the cellulose content was determined by the chlorination method of Cross and Bevan (an improved form of apparatus is described), the pentosans by Tollens' furfural method, moisture by drying at 105° C., ash by carbonisation with ammonium nitrate, and the lignin content by difference. In different samples of air-dried reeds the stems (57.09–60.92% of the total), sheaths (16.35–16.38%), and leaves (26.56–22.70%) contained respectively: cellulose, 38.64–40.93, 24.96–31.06, and 21.45–21.35%; pentosan, 17.27–19.66, 15.75–17.59, and 15.21–10.52%; lignin, 32.42–30.36, 37.42–35.35, and 42.01–50.79%; ash, 2.20–2.00, 14.00–8.00, and 11.33–10.67%; moisture, 9.47–7.00, 7.87–8.00, and 10.00–6.67%.—A. J. H.

*Swelling [of cellulose acetate]; Nature of the process of* —. E. Knoevenagel and O. Eberstadt. Kolloid-Chem. Beih., 1921, 13, 194–212.

CELLULOSE acetate does not swell in pure water or

in absolute alcohol, but it will swell readily in aqueous mixtures of alcohol, acetone, and acetic acid. In each case there is a definite mixture where the swelling is at a maximum. Unswollen cellulose acetate is only slowly dyed at 25° C. by a 0.05% aqueous solution of Methylene Blue, and the maximum coloration is reached only after several months. Under identical conditions swollen cellulose acetate is dyed to the maximum tint in a few minutes. Unswollen cellulose acetate is hydrolysed by aqueous alkali hydroxide solutions with the greatest difficulty, but the swollen material is completely hydrolysed in 30 mins. by a  $N/2$  solution of potassium hydroxide at 25° C. (Cf. J.C.S., June.) —J. F. S.

*Sulphite liquors; Titration of* —. R. Sieber. Paper, Apr. 6, 1921, 24–27.

Results are given of a comparative investigation of methods for analysing sulphite liquors, and the high values (5–15%) obtained for the free acid when determined by titration with standard alkalis are shown to be due to carbon dioxide. For works practice the total acid is most suitably determined by means of the iodometric method. Sander's new method (J., 1921, 256 A) which depends on the reaction between bisulphite compounds and an excess of mercuric chloride, whereby hydrochloric acid is liberated, gives correct results only when carefully carried out and is therefore more suitable for laboratory control and for exhaustive investigations. The free acid is most conveniently determined by Sander's older method or by Höhn's method. This latter gives more accurate results (maximum error 1–2%) if methyl orange or *p*-nitrophenol be used as an indicator, instead of phenolphthalein.—A. J. H.

*Sulphite-pulp; Determination of the degree of digestion of* —. R. Sieber. Paper, Apr. 6, 1921, 17–22.

The degree of digestion of sulphite pulp may be estimated by means of a solution of bleaching powder. 21 g. of moist pulp (5 g. of dry pulp) is placed in a brown glass bottle (light appreciably affects the reaction) with 150 c.c. of distilled water and is allowed to stand for  $\frac{1}{2}$  hr. in the case of a dry pulp. 100 c.c. of a solution of bleaching powder containing 6% (on weight of pulp) of active chlorine is added, and after 1 hr. the mixture is filtered through a bronze sieve and the remaining chlorine is determined in 50 c.c. of the filtrate. The amount of chlorine absorbed by the pulp ("chlorine number") is proportional to its lignin content and is related to the quality of the pulp as judged by its physical properties. The results obtained by this method with hard and soft pulps are similar to those obtained by the methods of Klason (J., 1911, 79), Richter (J., 1912, 869), and Becker (J., 1920, 482 A). The chlorine numbers of 25 pulps varied from 2.7 to 8.5, but were found to be somewhat too high in the case of very completely digested pulps. The chlorine number is influenced by the poor washing of a pulp, but this may be avoided by washing the pulp with 150 c.c. of warm water for  $\frac{1}{2}$  hr.—A. J. H.

*Paper pulp; Dyeing of* —. J. Huehner. J. Soc. Dyers and Col., 1921, 37, 139–145.

In the manufacture of paper pulp the degree of disintegration of the fibres produced by beating greatly influences the affinity of the pulp for dyestuffs. When samples of pulp produced from purified cotton yarn were immersed under comparable conditions in a solution of Night Blue the amount of dyestuff absorbed was inversely proportional to the average length of the fibres, and while unbeaten cotton yarn showed a constantly increasing absorption during 72 hrs., the absorption by the most highly disin-



tegrated pulp was almost complete during the first hour. The highly disintegrated pulp absorbed twice as much dyestuff as the unbeaten yarn, but had no greater depth of shade. In the case of pulp made from wool, increased disintegration augmented the rate of absorption but did not influence the amount of dyestuff absorbed. When immersed in a solution of Night Blue, jute, bleached poplar cellulose, and unbleached sulphite-cellulose absorbed the dye more quickly than bleached esparto cellulose and cotton half-stuff. Purification of sulphite-cellulose reduced its affinity for the dyestuff. The sizing of paper greatly increases its affinity for dyestuffs, and when resin size was added to a dye-bath containing Metanil Yellow or Scarlet R and alum, the pulp immersed therein rapidly absorbed 80–90% of the dyestuff. For the production of cheaper and faster shades, such mineral colours as Iron Buff, Chrome Yellow, Manganese Bronze, and Prussian Blue should be precipitated within the pulp. Acid dyestuffs have small affinity for paper unless it is heavily sized. Fast shades are obtained by precipitating an acid dyestuff by means of a basic dyestuff within paper pulp, and useful combinations are Auramino and Naphthol Yellow S; Chrysoidine, Magenta, or Brilliant Green and Orange II.; Victoria Blue and Water Blue or Methylene Blue, Rhodamine and Eosin, etc. Direct dyes are especially suitable for the production of mottled paper, and the shades obtained are fast.—A. J. H.

*Straw pulp for the manufacture of cardboard; Colouring* —. H. Press. Papierfabr., 1921, 19, 261–268, 365–368.

THE possible production of coloured cardboard by the direct addition of mineral or organic colouring matters to the straw pulp in the hollander was investigated. The dried straw pulp (after previous boiling with milk of lime under 4–5 atm. pressure) contained cellulose 40.4%, pentosans 20.5%, lignin (by diff.) 29.9%, and ash 9.2%. The mineral matter consisted chiefly of calcium silicate,  $\text{CaSiO}_3$ , and this was not removed in the preparatory treatment of the pulp. A solution of the dyestuff was added to a known weight of pulp, and after 20 mins. the exhausted dye solution was poured into a glass cylinder and its colour matched by the addition of a standard solution of the dye to water (which had been poured off from undyed straw pulp) contained in another cylinder. Straw pulp absorbed 95% (of the dyestuff added) of direct cotton dyes, 78–93% of basic dyes, and 53–75% of acid dyestuffs. The absorption of mineral colouring matters was determined by means of ash estimations and varied from 17 to 54%. For the production of a khaki-coloured cardboard a mixture of a mineral and an organic colouring matter was found to be suitable.—A. J. H.

*Paper; Investigations of materials suggested for the manufacture of* —. Bull. Imp. Inst., 1920, 18, 323–335.

STEMS of *Cyperus Papyrus* from Egypt treated with caustic soda under conditions similar to those used for the preparation of paper pulp gave 34–42% of dry pulp, which was difficult to bleach and was unsuitable for the production of white paper. Samples of Bourdie grass (*Typha* sp.) from Egypt gave about 35% of dry pulp, which was difficult to bleach, and produced a harsh, stiff paper, which shrunk considerably in drying. The Kokerboom (*Aloe dichotoma*) from South Africa is not a suitable paper-making material, and is inferior to other South African products. Van stems (*Hibiscus tiliaceus*) from Fiji gave a good yield of pulp, but of inferior quality. Matai wood (*Podocarpus spicatus*) from New Zealand gave a very low yield of pulp, which was not suitable as a paper-making material. "Tabocca Brava," a bamboo from Brazil, furnished, on rather drastic treatment, a good yield

of pulp of fairly high quality, suitable for wrapping papers. Aninga (*Montrichardia arborescens*) stems from Brazil yielded a pulp furnishing a good brown paper, but the pulp could not be bleached sufficiently for the manufacture of paper suitable for writing or printing purposes.—J. C. K.

*Carnauba wax palm*. Grimme. See XII.

*Cellobiose*. Karrer and Widmer. See XVII.

*Maize cob cellulose*. Marsh. See XXII.

#### PATENTS.

*Pressed [fibrous] material; Manufacture of articles from* —. Ottmar Reich, and H. Weiss. E.P., 133,952, 13.10.19. Conv., 23.3.17.

DRIED straw and similar reed-like materials are disintegrated, treated with dilute solutions of alkalis (e.g., 0.5% of caustic soda), acids (e.g., 0.42% of nitric acid), or sulphites, whereby ligneous incrusting substances are removed, and are then bleached. Successive treatments of the raw material with caustic soda and a hypochlorite give better results than when both substances are used together. The moist pulp so obtained is "formed" under pressure in moulds made of porous material, which allow excess of water to be expressed.—A. J. H.

*Balloon envelope material and process of manufacturing same*. Ballonhüllen Ges. E.P. (A) 139,795 and (B) 139,807, 3.3 and 4.3.20. Conv., 3.3.16 and 22.9.17.

(A) GAS-TIGHT balloon fabric is built up of a textile fabric united to an animal entrail skin, such as goldbeater's skin, by means of an adhesive layer containing glue and a substance to render it elastic. A suitable adhesive composition consists of 100 pts. of water, 10 pts. of gelatin, 10 pts. of Turkey-red oil, and 5 pts. of glycerin; 0.25 pt. (or 1%) of potassium bichromate or formaldehyde is added for waterproofing purposes. (a) The goldbeater's skin may be dispensed with if the textile fabric is coated with several layers of the adhesive composition. (Reference is directed in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 12,230 of 1890; J., 1891, 1016.)

—A. J. H.

*Degumming textile materials*. Soc. Suisse de Ferments. E.P. 145,583, 29.6.20. Conv., 30.6.19. (Cf. Tagliani, J., 1921, 256 a.)

THE rapid loss of diastatic power which occurs when a dilute solution of animal amylase (obtained from slaughterhouse waste) is heated at 55°–60° C., is greatly diminished by the addition of neutral salts (notably chlorides) of alkali or alkaline-earth metals. Sodium chloride and calcium chloride are suitable, 3–5 kg. being used per 1000 l. of solution. Textile materials are more easily penetrated by the solution of amylase if it contains a binary salt such as is present in pancreatic juice and purified ox gall.

—A. J. H.

*[Textile] fibres; Process and apparatus for degumming and washing* —. R. L. Pritchard. E.P. 161,219, 7.11.19.

FIBRES are packed in receptacles (7–15 cm. deep) having solid lateral walls and perforated bottoms, which are piled one above the other (the top one having a perforated cover) and secured so as to form a rigid column. This is fitted tightly within a vessel open at both ends and standing on a perforated shelf which rests on the bottom of a larger vessel containing the liquid used for treating the fibres. The liquid rises upwards through the fibre receptacles and is pumped back into the outer vessel. The fibres are thus subjected to a continuous unidirectional flow of liquor, whereby adhering

matters are loosened. The pile of fibre receptacles is then removed and made to slide slowly down through a vessel containing pure water, whereby the fibres are washed free from the loosened impurities.

—A. J. H.

*Retting of fibrous straw; Method of and bath for* — J. M. Masson. U.S.P. 1,374,941, 19.4.21. Appl., 12.1.20.

STRAW is steeped and then boiled in a solution containing trisodium phosphate and sulphonated oils, and is then washed.—A. J. H.

*Viscose silk; Manufacture of artificial* — E. Bronnert. U.S.P. 1,374,718, 12.4.21. Appl., 8.9.20.

ARTIFICIAL silk thread up to 1 denier in fineness is produced by spinning raw viscose, containing alkaline condensation products of phenols and aldehydes, in a bath containing a bisulphite, the concentration of which is inversely proportional to the fineness of the thread.—A. J. H.

*Cellulose; Manufacture of* — Badische Anilin- und Soda-Fahr. G.P. 331,950, 13.1.18.

WOOD, straw, or the like is treated with alkali and carbon dioxide. The treatments may take place once or several times, and in any succession. If ammonia is used as alkali it can be recovered as well as the carbon dioxide. After the aqueous liquors have been separated from the cellulose they can be concentrated in order to convert them into a fodder which, like molasses, is mixed with bran, peat, and the like.—C. A. C.

*Paper in pulp form; Sizing of* — J. W. Zanders. G.P. 303,341, 20.9.16.

MONTAN wax is saponified with caustic alkali, especially with potassium hydroxide, and is converted into a stable emulsion. The emulsion is added to the pulp in the hollander, and the montan wax is precipitated by aluminium salts or the like and fixed in the paper fibre. By sizing further than to the standard hardness a dense watertight paper is obtained without special impregnation.—C. A. C.

*Paper; Method for sizing — with animal glue or proteins*. F. Hassler. G.P. 331,350, 31.3.14.

THE precipitation of the glue and proteins is effected by means of sulphonic acids of unsaturated hydrocarbons, especially of the higher hydrocarbons of coal tar, or by condensation products of the sulphonic acids obtained by heating a mixture of them, or by condensing them with formaldehyde or with phenolsulphonic acids. As the precipitates may be easily obtained pure white and are not coloured by iron salts, they may be applied to all pulp products. The pulp is treated either alternately with the glue or protein solutions (the latter from yeast or fish) and the solutions of the precipitating agents, or glue (protein) and precipitant are added together dissolved in sodium carbonate; precipitation takes place when acid or aluminium sulphate is added. For surface sizing the paper is passed first through a solution of the condensation product and then through a solution of glue and afterwards is dried as usual. Examples of the application of the condensation products prepared from naphthalenesulphonic acid alone or with formaldehyde or phenolsulphonic acid are given.—C. A. C.

*Paper and pulp products; Method for sizing and impregnating* — H. T. Böhme, A.-G. G.P. 331,742, 16.5.18.

SULPHITE-CELLULOSE waste lyes are applied together with emulsions of coumarone-resin and animal glue, or coumarone-resin and crude montan wax, or

crude montan wax, or crude "montan wax colloid." Even when neutralised the sulphite lye acts as a precipitant for the above emulsions and participates in the sizing. For the precipitation of strongly alkaline emulsions acid sulphite-cellulose lye is used, or if the light-coloured neutral waste lye is used, other acid precipitants, as aluminium sulphate or dilute acids, are added. When precipitating emulsions of coumarone-resin and animal glue by sulphite lye the greater part of the glue is also precipitated. For the manufacture of water-proof paper or pulp products the dyed material is first treated with sulphite-cellulose lye, which may have to be acidified, and then is treated with the emulsions. The two-bath system may also be used with advantage for other textile products.—C. A. C.

*Furnace plant for the recovery of salts, especially in sodium sulphate cellulose factories*. W. Schacht. G.P. 317,082, 11.10.18.

A CALCINING and melting furnace mounted to rotate on wheels, rollers, or ball bearings, is movable parallel and perpendicular to the longitudinal axis of a rotary furnace, and the discharge outlet of the latter is faced with refractory material and adapted to fit into the inlet of the melting furnace, so as to make an almost air-tight junction for the transfer of material from the rotary furnace to the hearth of the melting furnace.—J. H. L.

*Sulphate-cellulose plants; Treatment of gases and vapours from* — A. E. Nielsen. G.P. 333,031, 6.3.19. Conv., 13.10 and 15.12.17.

THE gases are treated with an absorption mass containing calcium carbonate. The green waste lime from the manufacture of sulphate-cellulose may be used with advantage. After the treatment with the gases the mass contains: sodium carbonate, sodium sulphide, sodium sulphate, sodium thiosulphate, hydrogen sulphide, thiocyanic acid esters, sodium thiocyanate, acetone, other ketones, aldehydes and mercaptans, calcium sulphite, calcium sulphate, calcium sulphide, calcium oxide, calcium tetrathionate, calcium carbide, etc., which can be converted into useful products. Calcium thiosulphate is converted into sodium thiosulphate.—C. A. C.

*Fibres; Treating* — J. H. Pickup and G. A. Wilson. E.P. 161,600, 15.9.19.

SEE U.S.P. 1,350,621 of 1920; J., 1920, 653 A.

*Cellulose; Process for converting* — R. A. Kocher. U.S.P. 1,374,928, 19.4.21. Appl., 21.3.17.

SEE E.P. 107,219 of 1916; J., 1917, 973.

*Nitrocellulose and other esters; Process of reducing the viscosity of viscous solutions of* — O. Carlsson and E. Thall, Assrs. to Atlas Powder Co. U.S.P. 1,375,208, 19.4.21. Appl., 4.12.19.

SEE E.P. 136,141 of 1919; J., 1921, 296 A.

*Paper; Method and means for imparting a polished textile or like surface to* — Eastern Manufacturing Co., Assees. of H. J. Guild. E.P. 142,117, 21.4.20. Conv., 9.12.16.

SEE U.S.P. 1,277,714 of 1918; J., 1918, 687 A.

*Paper making [machines; Device for carrying reel of paper from drying rollers to calendering rollers of —]* Great Northern Paper Co., Assees. of C. E. Pope. E.P. 141,022 and 151,623, 28.1.20. Conv., 28.3.19.

See also pages (A) 399, *Coating composition* (U.S.P. 1,358,914). 405, *Sugar from wood* (E.P. 142,480); *Saccharification of cellulose* (E.P. 143,860); *Glucose from wood* (E.P. 143,212); *Fermentation of cellulose* (E.P. 161,294); *Fermentation process* (E.P. 161,870).

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Woollen fabrics; Two- and multi-coloured effects in* — M. König. *Textilber.*, 1921, 2, 178.

COLOURED effects may be obtained in the piece-dyeing of woollen fabrics by suitably treating part of the yarn before weaving. Yarn which has been treated with chlorine or caustic soda has an increased affinity for acid, mordant, and direct cotton dyes, and yarn mordanted with tannic acid and tin or antimony salts loses its affinity for acid wool dyes but is strongly coloured by basic dyestuffs. After treatment with solutions of potassium, ammonium, or calcium thiocyanate, wool has a permanently increased affinity for acid, afterchrome, and mordant dyestuffs. Wool which has been immersed for 1–1½ hrs. at 50° C. in a solution containing 25% of sodium thiosulphate and 16% (on weight of wool) of hydrochloric acid of 22° B. (sp. gr. 1.18) has a diminished affinity for acid, substantive, and mordant dyes, and an increased affinity for basic and vat dyestuffs.—A. J. H.

*Dyeing of wool; Direct* — L. Guglielmelli and C. G. Estrella. *Anal. Soc. Quím. Argentina*, 1920, 8, 325–326.

Wool and silk can be dyed directly by diazotisation and subsequent treatment with certain polyphenole and phenolic glucosides. By the use of metallic salts, such as potassium bichromate and copper sulphate, variations in shade and greater fastness to light can be obtained.—G. W. R.

*[Printing] reserves under Indigo; Function of manganese oxide in* — R. Haller. *Textilber.*, 1921, 2, 173–174.

THE conditions under which manganese compounds may resist the fixation of vat dyestuffs on fabrics have been investigated by means of a Schleicher-Schull dialysing cell, the material of which has been considered as having the properties of a cotton fibre. The dialyser was filled with a solution of manganese chloride and placed in a 2% solution of caustic soda. After 24 hrs. the inner liquid was clear and neutral, but a precipitate of manganese hydroxide had formed in the caustic soda solution. A cross-section of the thoroughly washed dialyser showed that a brown deposit of manganese hydroxide had formed on the outer half of the membrane, while the inner half was free from manganese. When a solution of indigo reduced by means of hydrosulphite was dialysed in the same dialyser, which was immersed in water, the indigo penetrated considerably into the deposit of manganese hydroxide. In another experiment, the dialyser (immersed in 2% caustic soda) contained a solution of 20 pts. of manganese chloride and 1 pt. of sodium bichromate, and after 24 hrs., no bichromate remained in the inner solution. A cross-section of the dialyser showed (inside to outside), a brown deposit of a manganese-chromium compound, a colourless layer, another brown manganese-chromium deposit, a broad colourless layer, and a small deposit of manganese hydroxide. These facts are attributed to the formation of a compound,  $2\text{MnO}_2 \cdot \text{Cr}_2\text{O}_3$ , which has been prepared, and is regarded as an adsorption compound of a manganese chromate with manganite. When the same dialyser was used for the dialysis of a solution of indigo and hydrosulphite, the indigo only penetrated as far as the first manganese-chromium deposit. It is concluded that indigo reserve pastes which contain manganese chloride and sodium bichromate are effective because of the high oxidising power of the manganese chromate which is formed, and that for this type of reserve indigo vats prepared with zinc and lime are

more suitable than those containing hydrosulphite, since the latter diffuse more quickly through the dialysing membrane.—A. J. H.

*Ultramarine; Printing fabrics with* — G. Stein. *Textilber.*, 1921, 2, 176–177.

It is difficult to obtain effects which are fast to washing by means of printing pastes which contain starch and coarse varieties of ultramarine, since the affinity of ultramarine for cotton decreases with an increase in the size of the ultramarine particles. Moreover, in order to avoid "scumming," it is necessary to add to the printing paste such substances as dextrin, gums, and caustic soda, and these decrease the affinity of ultramarine for the fibre.—A. J. H.

### PATENTS.

*Bleaching of fabrics.* J. Kershaw. E.P. 162,198, 19.5.20.

Wool, cotton, silk, and other fabrics in piece form are passed through a bath containing a cold 25% solution of hydrogen peroxide made slightly alkaline with ammonia, and after a period (16 hrs. for medium weight woollens) are washed with soap, and then passed through a cold 8½% solution of sodium hydrosulphite (a trace of sulphuric acid may sometimes be added), washed, and dried. The bleaching effect is permanent and the baths may be used continuously.—A. J. H.

*Dyeing with acid sulpho-amino dyestuffs; Process for* —, and *manufacture of lakes.* Farbenfabr. vorm. F. Bayer und Co. E.P. 143,242, 12.5.20. Conv., 31.10.14.

DYINGS and lakes produced from acid dyestuffs which contain both sulpho- and amino-groups are rendered faster to light by treatment with a complex metatungstic acid or its salt. Phospho-tungstic, silico-tungstic, and antimony-tungstic acids are suitable. In wool dyeing, 1% of the tungstic acid is added to the dye-bath before or towards the end of the dyeing process. In the manufacture of lakes, the tungstic acid is added to a solution of the dyestuff, and barium chloride or other suitable precipitant is added.—A. J. H.

*Dyeing tops, yarn and the like; Apparatus for* — A. Ashworth. U.S.P. 1,374,623, 12.4.21. Appl., 15.10.20.

A PERFORATED tube is mounted vertically within a dyeing vat, and means are provided for forcing a dye or mordant liquor through the tube and a series of collectively collapsible tubular yarn spools which are mounted upon it.—A. J. H.

*Warp-dyeing machine.* W. F. Haskell. U.S.P. 1,375,339, 19.4.21. Appl., 4.4.18. Renewed 15.9.20.

WARP yarn is drawn through a series of unit coils each having inlets for the dye liquor, which is supplied to the last unit. The overflow for exhausted liquor from the last unit is connected with the inlet of one of the preceding units.—A. J. H.

*Silks; Process of treating [dyeing grege]* — E. L. Maupai. E.P. 161,625, 10.12.19.

See U.S.P. 1,332,675 of 1920; J., 1920, 514 A.

*Dyeing paper pulp.* Huebner. See V.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Nitric acid; Concentration of* — E. Galle. Z. angew. Chem., 1921, 34, 168–170, 173–175.

NITRIC acid prepared by the oxidation of atmospheric nitrogen or of ammonia always has to be concentrated, the highest initial strength obtained

being 50%  $\text{HNO}_3$ . The Pauling tower as improved by Hoening is used for this purpose. The tower, about 8 m. high, is made of cast iron rings lined with acid-resistant material, and packed with Raschig rings carried on silicon-iron grids interposed between adjacent sections. The uppermost section, which is not packed, includes a stoneware acid distributor, a gas-exit pipe to the nitric acid cooler, acid-feed pipes, and thermometer. The bottom section is fitted with a manhole, steam-jet, and run-off pipe for dilute denitrated sulphuric acid. The nitric acid cooler is a worm of silicon-iron, and any uncondensed gases are drawn by a stoneware injector to an absorbing tower, the weak acid from which is returned to the process. The acids are elevated by silicon-iron pumps with chromium-steel shafts. The process is started by feeding with concentrated sulphuric acid and blowing in steam until the temperature at the top reaches  $130^\circ\text{C}$ . In normal working the temperature at the top of the tower stands at about  $112^\circ\text{C}$ . The quantity of sulphuric acid required depends on the dilution of the nitric acid. With the usual nitric acid of 1.26 sp. gr., 5 pts. of sulphuric acid to 1 pt. of nitric acid is required. The sulphuric acid may be replaced by an equivalent quantity of the same in the form of mixed waste-acid. In the case of nitric acid containing hydrochloric acid the consumption of sulphuric acid is greater. Laboratory experiments showed that, with an effective fractionating column, dilute nitric acid can be concentrated up to 65%  $\text{HNO}_3$ , without the use of sulphuric acid with no appreciable loss. To complete the concentration of this acid 2 pts. of sulphuric acid (monohydrate) is required for each 1 pt. of water present in the nitric acid. The large-scale application of this method would effect a great increase in the capacity of a given plant and saving in sulphuric acid. The addition of hydrochloric acid to the mixed acids greatly raises the boiling point, necessitating more sulphuric acid, and in addition the latter must be in excess to prevent formation of nitrosyl chloride. In the case of mixed waste acids about 50% of the  $\text{N}_2\text{O}_4$  present is oxidised during distillation, but absorption in alkali is advisable to recover the remainder.—C. I.

*Electrolytic cells; Marsh* — for chlorine, caustic soda, and hydrogen. C. W. Marsh. Trans. Amer. Electrochem. Soc., 1921, 297–306. [Advance copy.]

THE Marsh electrolytic cell comprises a top of stoneware or other suitable material shaped like an inverted trough with ends extended downwards. The cell is suspended from this, or supported in a box, and from the top are also suspended the anodes, whilst the cathode is clamped to the top and ends. Openings are provided in the top for brine inlet, brine control device, brine level indicator, brine gauge glass, chlorine outlet, and for cleaning purposes. The anodes consist of graphite rods, 4.4 cm. diam. and 60 cm. long, arranged one above the other with spaces between, and are fixed to a rectangular graphite section. The cathode of perforated sheet steel is corrugated to conform to the surface of the horizontal graphite rods, and an asbestos paper diaphragm is tightly clamped to the edges of the cathode. The cathode with the ends and top forms the electrolytic compartment; it may be confined if the hydrogen gas is to be recovered. For small industrial plants and water and sewage purification, batteries of 100–300 amps. are suitable; the cells measure  $25 \times 72 \times 80$  cm. and a battery of 15 cells yields 5.5 kg. of chlorine, 6.1 kg. of caustic soda, and 0.15 kg. of hydrogen per hour. Normally, the life of the batteries is six months and of the anodes two years. The cost of chlorine from such a plant, taking into account the value of the caustic soda, is 4.5–5.0 cents per lb., as compared with liquid

chlorine at 12 cents per lb. For large electrolytic alkali, bleach, and chlorine plants, batteries of 1250 to 5000 amps. are used. The cells measure  $25 \times 134$  cm. and have a voltage of 3.35 and power efficiency of 63%. The batteries can be operated at double capacity, the current available then being 2500–10,000 amps. In this case the cells measure 25 cm.  $\times$  5 m.; the voltage will be 4, and the power efficiency 50%. Over long periods the ampere efficiency amounts to approximately 90%; it may reach 95–97%. Features of the Marsh cell are the low average voltage resulting from the provision of a large electrode surface, regular distribution of deposits on the diaphragms preventing clogging of the passages, good circulation, and effective removal of chlorine bubbles. Small cells of large capacities operating at high efficiencies can thus be successfully employed.—W. J. W.

*Ammonia synthesis equilibrium; Interpretations of* —. R. S. Tour. J. Ind. Eng. Chem., 1921, 13, 298–300.

EQUILIBRIUM in the reaction  $\frac{1}{2}\text{N}_2 + \frac{3}{2}\text{H}_2 \rightleftharpoons \text{NH}_3$  may be expressed by the formula  $a/(1-a-c)^2 = KP(r^{3/2})/(1+r)^2$  where  $a$ =volume fraction of ammonia at equilibrium,  $c$ =volume fraction of inert gases at equilibrium,  $r$ =volume ratio of hydrogen to nitrogen at equilibrium,  $P$ =total pressure in atm., and  $K$  is a function of the absolute temperature. A set of curves shows the effect on the ammonia content at equilibrium of a variation of any one of the conditions when the others have the arbitrary values:  $T=773^\circ\text{abs.}$ ,  $P=100$  atm.,  $r=3$ ,  $c=0$ . Both curves and equation show that effect of temperature is very marked; a reduction from  $500^\circ$  to  $485^\circ\text{C}$ . is as advantageous as a rise in pressure from 100 to 120 atm. Pressure increases the ammonia content at a decreasing rate with increasing ammonia content. Changes in the ratio of hydrogen to nitrogen have a small effect; a variation from the theoretical to the ratio  $2\text{N}_2:3\text{H}_2$ , or to  $0.5\text{N}_2:3\text{H}_2$ , involves a reduction of less than 10% of the equilibrium content. The effect of inert diluents is not only to lower the partial pressures of the reacting gases, but to dilute these as well. Each of the terms of the equation may be plotted as a separate curve for the sake of simplicity in computation, and a simple nomograph has been constructed for determining the equilibrium ammonia content when only temperature and pressure are variable,  $r$  has its theoretical value of 3.0, and  $c=0$ .—W. J. W.

*Hydrogen for the synthesis of ammonia; Manufacture of* —. G. Claude. Comptes rend., 1921, 172, 974–977.

THE source of hydrogen suggested is water-gas, town gas, or coke-oven gas. In order to separate the hydrogen and the carbon monoxide the author suggests the use of solvents under pressure. By the use of ether under a pressure of 100 atm. and a temperature of about  $-50^\circ\text{C}$ . it is easily possible to obtain hydrogen containing less than 0.2% of carbon monoxide. This pressure should not be greatly exceeded, as above this limit the solubility of hydrogen in the solvent increases very rapidly in proportion to that of the carbon monoxide.—W. G.

*Potassium compounds; Application of the vapour pressures of* — to the study of the recovery of potash [from silicates] by volatilisation. D. D. Jackson and J. J. Morgan. J. Ind. Eng. Chem., 1921, 13, 292–295.

THE information gained from experiments on the vapour pressures of potassium compounds (J., 1921, 177 A) has been applied to an investigation of their volatilisation. If greensand is heated with lime to  $1300^\circ\text{C}$ ., up to 90% of the potash is volatilised, whereas in the case of feldspar not more than 20% is obtained, the explanation lying in the fact that the

water liberated from the former silicate assists the formation of potassium hydroxide which has a high vapour pressure. The efficiency of calcium chloride, in admixture with lime, as a volatilising agent, was tested with glauconite and felspar, in such proportions as to give a residue having the composition of Portland cement. In the case of glauconite, volatilisation was practically complete, even at 215° C.; with felspar it was less so. Contrary to Packman and Cornwell's claims (U.S.P. 1,202,327; 1,1916, 1221), it was found that when sufficient chloride is present in the mixture to form potassium chloride, no advantage is gained by introducing water vapour. From mixtures of greensand with limestone in smaller proportions than required for Portland cement, without addition of a chloride, volatilisation is very small up to 1170° C., even in presence of water vapour; but if a chloride is added, mixtures containing only one-third of limestone, heated just below their fusing point, show a high volatilisation of the potassium compound. As a volatilising agent sodium chloride appears to be superior to calcium chloride.—W. J. W.

*Potassium salts; Solubility of different — in mixtures of water and alcohol.* M. Pierrat. *Comptes rend.*, 1921, 172, 1041—1043.

THE solubility at 14° C. in aqueous alcohol of varying concentrations and the electrical conductivity of aqueous solutions containing the same weight of salt in a volume of water equal to that of the aqueous alcohol have been determined for the bicarbonate, perchlorate, platinichloride, fluosilicate, and cobaltinitrite of potassium. The solubilities, in g. per l., in alcohol of 94.7% concentration (by weight) at 14° C., of the different salts in the order given above are 0.05, 0.15, 0.02, 0.0096, 0.026.

—W. G.

*Sodium sulphide; Action of — on ferric oxide.* J. C. Witt. *J. Amer. Chem. Soc.*, 1921, 43, 734—740.

AN excess of sodium sulphide converts ferric oxide into a black amorphous substance, which after removal of most of the sodium sulphide dissolves in water to form a brilliant green solution. The solution is a reversible sol containing less than 0.07 g. of iron per litre. (*Cf. J.C.S.*, June).—J. F. S.

*Sodium silicate; Manufacture of —.* A. A. Percazzo. *Anal. Soc. Quim. Argentina*, 1920, 8, 404—409.

AN improved plant for the preparation of sodium silicate by fusion is described. The furnace is rectangular in plan and is divided into two unequal portions by a bridge which is so constructed that only fused material can pass under it while floating unmelted material is held back. When a fresh charge is delivered into the larger compartment the fused sodium silicate is forced under the bridge into the smaller compartment of the furnace and overflows into a tank which is maintained full of cold water. The sudden cooling of the fused silicate produces a fine granular product. The apparatus is continuous in working and the product obtained is more readily dissolved than that obtained by older methods involving the use of grinding machinery.

—G. W. R.

*Fluorides; Modification of Starck and Thorin's method for the determination of —.* E. D. Garcia. *Anal. Soc. Quim. Argentina*, 1920, 8, 321—324.

STARCK and Thorin's method (*J.*, 1912, 259) for the estimation of fluorides in aqueous solution is modified, the excess of calcium in the filtrate from the mixed precipitate of calcium oxalate and fluoride being estimated volumetrically. Knowing the amount of calcium required for the calcium

oxalate, the amount of calcium fluoride and, hence, the percentage of fluorine in the original solution is obtained. Insoluble fluorides are first fused with silica and sodium carbonate. After treatment of the mass with water, silica is removed by ammonium carbonate and fluorine estimated as above.

—G. W. R.

*Hypochlorous acid; Electrometric titration of —.* W. D. Treadwell. *Helv. Chim. Acta*, 1921, 4, 396—405.

WHEN hypochlorite is titrated with either arsenious acid or potassium iodide the end point may be determined accurately by electrometric means. The comparison electrode employed consists of a glass tube about 8 cm. in length, with its end drawn out to a capillary and turned up and the extremity of the capillary closed by a plug of filter paper or a drop of gelatin containing potassium sulphate. In this tube is placed potassium sulphate solution mixed with either a little hypochlorite solution already titrated or a drop of about *N*/100 iodine solution containing iodide. As electrodes use is made of two smooth platinum wires, one dipping into the hypochlorite solution and the other fairly deeply into the comparison electrode. These two electrodes communicate by way of either a high-resistance millivoltmeter or a galvanometer with suitable resistance. A sudden drop of the galvanometer deflection to zero marks the end point. When arsenious acid solution is used, the alkaline hypochlorite should be neutralised and at once titrated, but the loss due to premature formation of chlorate is inappreciable if the titration is carried out in dilute solution, at the room temperature, and within a few minutes of neutralisation. The end point becomes indistinct if a solution containing alkali carbonate or hydroxide is titrated with arsenious acid. The optimal hydrogen-ion concentration for the end point of the titration of a hypochlorite with potassium iodide, calculated from the equilibrium,  $\text{IO}_3^- + 5\text{I}^- + 6\text{H}^+ \rightleftharpoons 3\text{H}_2\text{O} + 3\text{I}_2$ , is  $10^{-7}$ , which is that of the neutral point. The fundamental objection to this method is the pronounced sensitiveness of the end point to the reaction, and Pontius (*J.*, 1904, 133), who devised the method, made use of a highly alkaline solution [ $(\text{H}^+) = 10^{-12}$ ], which retards the end point. Good results are obtained with a solution alkaline with bicarbonate if the available chlorine amounts to about 0.1 equivalent per litre, but *N*/100 hypochlorite solution yields unreliable values under these conditions. When a very dilute solution is titrated, the final stages must be carried out very slowly, otherwise the blue coloration of the starch appears too late.

—T. H. P.

*Hydrocyanic acid; Formation of — in plants.* P. Monaul. *J. Biol. Chem.*, 1921, 46, 297.

THE formation of hydrocyanic acid was observed *in vitro* in sunlight, in a solution containing potassium nitrate and formaldehyde, but only if the reaction was acid to methyl orange. It is suggested that hydrocyanic acid may thus be formed in plants.—G. B.

*Hydrofluosilicic acid; Equilibria of —.* L. J. Hudleston and H. Bassett. *Chem. Soc. Trans.*, 1921, 119, 403—416.

THE standard "Analytical Reagent" methods for the detection of silica in hydrofluoric acid are inadequate. The proportion of hydrofluosilicic acid in mixtures with hydrofluoric acid can be determined by complete neutralisation with sodium hydroxide (*cf. Wagner and Ross, J. Ind. Eng. Chem.*, 1917, 9, 1116), according to the equation,  $\text{H}_2\text{SiF}_6 + 6\text{NaOH} = 6\text{NaF} + \text{H}_2\text{SiO}_3 + 3\text{H}_2\text{O}$ . The estimation is carried out in conical flasks of wax or waxed glass, closed with rubber stoppers and con-

taining measured quantities of sodium hydroxide coloured with phenolphthalein. Weighed quantities of the mixed acids are placed in wax test tubes inside the flasks and the contents of both mixed by shaking. When the colour has faded (the time being measured), further alkali is run in until permanent neutralisation is reached. If  $n$  c.c. of alkali were originally present and  $N$  c.c. were required for total neutralisation,  $(N-n)/N \times 100$  is the percentage of total non-available hydron (i.e., the "complex" hydrofluosilicic acid) in the solution at the moment of fading. From the different values for this figure a value for the initial concentration of the complex, i.e., the percentage of hydrofluosilicic acid initially present in the mixture, can be calculated. For concentrated solutions the measurement of  $(N-n)/N \times 100$  is accurate to 0.25%, but for dilute solutions only to 1%. The time readings, due to the difficulty of bringing about complete mixing, and measurement of temperature due to heat of reaction, are also liable to error. The rate of dissociation of sodium silicofluoride into silicon tetrafluoride and sodium fluoride has been determined, together with the formula for the variation of the velocity constant with temperature. The equilibria set up when silicon tetrafluoride is passed into water are discussed. All four substances—hydrofluosilicic acid, hydrofluoric acid, silicic acid, and unchanged silicon tetrafluoride—are present, though in small concentration. Silicic acid is shown to exist in true solution to the extent of at least 0.003 mol. per litre at 15° C.—P. V. M.

*Hydrogen peroxide; Kinetics of the decomposition of* — F. Bürki and F. Schaaf. *Helv. Chim. Acta*, 1921, 4, 418–425.

DECOMPOSITION of hydrogen peroxide in alkaline solution is a reaction of the first order, the velocity of the reaction being dependent on the concentration of the base but independent of its nature and conditioned apparently by the concentration of the hydroxyl ion alone.—T. H. P.

*Carbon; Adsorption by* — H. Herbst. *Biochem. Zeits.*, 1921, 115, 207–219.

THE adsorptive power of charcoal depends partly on the content of pure amorphous carbon, and can in some cases be increased by removing tarry impurities by means of a solvent or by distillation, or by destroying them by chemical means, as in the Aussig zinc chloride process. At about 1150° C. charcoal is fairly rapidly transformed into graphite and becomes a less powerful adsorbent. The author has prepared pure amorphous carbon of maximal adsorptive power, and expresses the "true" activity of technical samples as percentages of the former (comparison by CO<sub>2</sub> isotherm). Experiments with gaseous chloropicrin, benzyl chloride, and phosphorus tribromide, lead to the conclusion that in each case 1 mol. is fixed by 6 atoms of pure carbon, probably present as a C<sub>6</sub>-ring.—G. B.

*Iodic acid.* Hendrixson. See XXIII.

*Hydrochloric, hydrobromic, and hydriodic acids.* Longinescu and Chaborski. See XXIII.

*Nitric acid.* Longinescu and Chaborski. See XXIII.

#### PATENTS.

*Sulphuric acid, Concentration of* — in a vacuum. Badische Anilin- u. Soda-Fabr. G.P. 302,553, 12.1.17.

A HEATING chamber is provided with pipes of perforated sheet iron coated internally with lead which is connected homogeneously to the iron at the perforations. The exterior of the pipes may also be covered with lead. These pipes resist the chemical action of the boiling liquid and also the difference

of pressure. The chamber is also lined with lead, and a continuous process is practicable.—C. I.

*Ammonia oxidation; Process for the activation of contact masses for catalytic* — Badische Anilin- u. Soda-Fabr. G.P. 304,232, 27.1.15.

THE activating material is applied to the catalyst in a state of fine division in the reaction chamber. This method may be applied to the revival of old contact masses without interference with the process. In the case of a contact mass containing bismuth, an acid solution of bismuth nitrate containing 10% Bi is injected into the reaction chamber.—C. I.

*Sulphate kilns; Apparatus for the introduction of sulphuric acid into mechanical* — Farbenfabr. vorm. F. Bayer und Co. G.P. 332,954, 29.11.19. Addn. to 331,238 (J., 1921, 301a).

IN place of an acid feed vessel, a pipe secured to the rotating spindle and curved at the end is used.—C. I.

*Sulphur dioxide; Obtaining and utilising* — from blast-furnace slag. L. H. Diehl. E.P. 139,172 16.2.20. Conv., 16.1.17.

FOR the recovery of sulphur, existing as sulphide from blast-furnace slag, a current of air, preferably heated, is blown through, or led over the slag in sufficient amount to give a gas mixture containing not less than 2% SO<sub>2</sub>, whilst at the same time avoiding solidification of the slag or further oxidation of its contained metallic oxides. The operation may be conducted in a slag pan with hood and gas outlet, or in a covered sump inserted in the slag run. In conjunction with the air oxidation, the introduction of oxidising compounds, such as calcium sulphate, is recommended, these being added in a highly heated or molten condition. The sulphur dioxide recovered is free from arsenic (Reference is directed, in pursuance of Sect. 7 Sub-sect. 4, of the Patents and Designs Acts, 190 and 1919, to E.P. 11,544 of 1899 and 2405 of 1908 J., 1899, 444; 1908, 1209.)—W. J. W.

*Percarbonates; Manufacture of* — Deutsche Gold- u. Silber-Scheide Anstalt vorm. Rössler. E.P. 117,085, 24.6.18. Conv., 25.6.17.

ALKALI percarbonates mixed with carbonates are prepared by interaction of alkali peroxide and bicarbonate in presence of a limited amount of water. The percarbonate is separated from crystallised carbonate and salted out by addition of sodium chloride. Negative catalysts such as alkali silicates, stannic acid, or gums may be added to the percarbonate solution.—C. I.

*Iron salt [for tanning]; Production of a solid non-hygroscopic* — O. Röhm. E.P. 146,214, 26.6.20. Conv., 27.12.18.

BY the action of chlorine on ferrous sulphate, whether in the solid state or in solution, a non-hygroscopic compound of the composition FeSO<sub>4</sub>Cl is obtainable, which is specially suitable for tanning purposes.—D. F. T.

*Ammonia; Method of and means for use in manufacture of [aqueous]* — R. P. Douglas. E.P. 161,244, 31.12.19.

A PORTION of the vapours from the liming chamber of an ammonia still is passed through a reflux condenser to remove water, and thence to a finishing condenser, where strong aqueous ammonia is obtained.—C. I.

*Ammonia; Process for the preparation of* — from nitrogen. H. Hampel and R. Steinau. G.P. 310,761, 24.8.17.

A METAL is treated at high temperature and pressure with hydrogen and with ammonium chloride



or other ammonium compound or other substance which liberates nascent hydrogen. The ammonium or metallic salt used is recovered, as also is the metal, by reduction of the oxide or carbonate formed either directly or as a result of subsequent reactions.—C. I.

*Ammonia and ammonium sulphate; Production of — by the action of steam on cyanogen compounds formed in the distillation of organic nitrogen compounds.* F. J. Collin, A.-G. zur Verwertung von Brennstoffen und Metallen. G.P. 328,829, 18.5.19.

A MIXTURE of gas containing cyanogen and steam, after removal of free ammonia, is treated with excess of sulphuric acid, with continual heating, e.g., in a column apparatus, whereby the cyanogen is converted into ammonia, which combines with the acid to form ammonium sulphate. In the case of gases of low cyanogen content the cyanogen is absorbed in water or ammoniacal liquor, the resulting liquor is distilled, the ammonia absorbed, and the waste gases from the sulphate saturator treated as described. The reaction velocity is increased by the presence of hydrogen sulphide.—C. I.

*Ammonium bicarbonate; Preparation of dry — from wet precipitated salt.* Badische Anilin- u. Soda-Fabr. G.P. 301,674, 12.1.16.

THE salt is thoroughly exposed to a counter current of warm, indifferent gases (air, carbon dioxide, or flue-gases) which have passed through a drying chamber.—C. I.

*Sodium pyrophosphate; Manufacture of acid —.* A. Kelly. E.P. 161,273, 5.1.20.

THE calculated amount of acid, e.g., hydrochloric acid, is added to a strong solution of sodium pyrophosphate, and the acid salt precipitated by saturating with sodium chloride.—C. I.

*Phosphatic materials; Process of sintering —.* F. S. Washburn, Assr. to American Cyanamid Co. U.S.P. 1,373,471, 5.4.21. Appl., 23.7.20.

A MIXTURE of phosphatic and silicious materials is sintered to form a porous mass which is mixed with carbon and heated in an electric furnace, the evolved phosphorus being recovered.—W. J. W.

*Aluminous compounds; Manufacture of —.* P. Spence and Sons, Ltd., H. Spence, and W. B. Llewellyn. E.P. 161,606, 11.10.19.

THE process in which iron is precipitated from aluminium sulphate solution by addition of a potassium salt and 5–10% excess of alumina (E.P. 9148 of 1914; J., 1915, 799) is made more rapid by adding to the solution a proportion of impalpable mud. This is prepared by grinding the silicious residues from the treatment of aluminous minerals, or native alunite may be used. The basic ferric potassium sulphate is then readily separated from the liquor. Part of it is used for a fresh operation, and the valuable constituents in the mud are thus gradually concentrated and may be recovered.—C. I.

*Aluminium hydroxide; Manufacture of —.* M. Lindner. G.P. 333,383, 1.5.18.

STRONGLY cooled dilute solutions of aluminium salts are added to dilute and strongly cooled solutions of ammonia, which are kept vigorously stirred by means of a current of air saturated with ammonia. Aluminium hydroxide is thereby precipitated in a light gelatinous form (sp. gr. about 1) possessing a high adsorptive power.—A. R. P.

*[Aluminium sulphate;] Treatment of colloid-containing mediums [e.g., in ore flotation, and in manufacture of —].* W. A. Deane, Assr. to The Dorr Co. U.S.P. 1,359,037, 16.11.20. Appl., 13.1.20.

THE product obtained by the action of sulphuric acid on hauxite is treated with "packing-house stick" (an organic gel obtained as a by-product in the packing-house industry by boiling waste meat products etc. with water), whereby colloidal impurities (clay, silica, etc.) are coagulated and can be easily separated. Packing-house stick may also be used as a dispersing medium in ore flotation processes; the deflocculated colloids are separated from the ore pulp by decantation, and their interference in the subsequent flotation operation thus avoided.

*Copper sulphate; Process of obtaining — from metallic copper.* P. A. Mackay. E.P. 161,658, 9.1.20.

REFINED or blister copper is dissolved in hot 70% sulphuric acid in presence of silver nitrate or other salt of a metal more electronegative than copper. Blister copper often contains enough silver for the purpose, and in this case only nitric acid is added. Excess of metal is used, and the silver etc. is deposited on the residual copper.—C. I.

*Potassium carbonate; Method of making —.* E. P. Stevenson, Assr. to A. D. Little, Inc. U.S.P. 1,360,046, 23.11.20. Appl., 19.1.20.

A SOLUTION of potassium acetate in ethyl alcohol is treated with carbon dioxide, the potassium bicarbonate is separated, and the alcoholic solution is then treated with a reagent, e.g., lime, to produce a salt (calcium acetate) of low solubility, which is used in making further quantities of potassium acetate.—W. J. W.

*Potassium salts; Process for recovering — from solutions carrying borates or carbonates or both.* C. E. Dolbear, Assr. to D. C. Norcross, J. H. Miller, and G. J. Henry. U.S.P. 1,373,179, 29.3.21. Appl., 30.10.19.

SOLUTIONS of potassium salts containing boron compounds are treated with an aluminium compound, the insoluble aluminium borate is removed, and the potassium salt recovered from the residual solution.—W. J. W.

*Alkali sulphides; Process of making —.* W. H. Landers, Assr. to H. S. Loud. U.S.P. 1,374,209, 12.4.21. Appl., 17.11.19.

AN alkali salt of a volatile mineral acid is intimately mixed with "sludge-acid" containing at least 5% of carbon, and the mixture heated to reduce the sulphate formed to sulphide.—C. I.

*Carbides and derivatives thereof; Process and apparatus for manufacture of —.* F. E. Norton. U.S.P. 1,374,317, 12.4.21. Appl., 1.4.18.

IN a continuous process for the manufacture of carbides from a mixture of carbon and oxides or hydroxides, the necessary heat is supplied by combustion of part of the carbon with oxygen.—C. I.

*Basic melts rich in lime; Process for the preparation of —.* L. H. Diehl. G.P. 307,190, 21.12.17.

A MIXTURE of about molecular equivalents of calcium oxide and sulphate is melted with an easily fusible compound, such as sodium chloride, calcium chloride, sodium hydroxide, or sodium sulphate. Limestone may be used in place of lime. The melt can be used for the decomposition of sulphides in furnace slags by agitating them with a current of air and adding the basic mixture, preferably in a molten state. Sulphur dioxide is evolved by the



interaction of sulphide, sulphate, and air; the residual slag is granulated and the granular mass dried and powdered to the fineness of cement.—C. I.

*Hydrogen peroxide; Process of obtaining solid compounds of —.* E. Merck. G.P. 331,111, 29.3.12. Addn. to 303,680 (J., 1918, 371 A).

Hydrogen peroxide solutions rendered stable by the methods described in G.P. 174,190, 203,019, and 216,263 (J., 1908, 1204; 1909, 1314) are evaporated with neutral organic or inorganic carriers. Hydrogen peroxide is thus obtained in combination with the substance used in a solid stable state.—C. I.

*Hydrogen; Manufacture of — by dissolving iron in sulphuric acid.* R. Blum and M. Buchner. G.P. 303,281, 8.1.16.

Iron is treated with the equivalent quantity of sulphuric acid, with the application of heat or pressure, or both, for the production of hydrogen. The resulting ferrous sulphate is calcined in a current of air to ferric oxide with the evolution of sulphur trioxide, which is re-converted into sulphuric acid and used again in the process together with the iron resulting from the reduction of the ferric oxide.—A. R. P.

*Silica gels and processes of making same.* W. A. Patrick. E.P. 136,543, 6.12.19. Conv., 7.12.18.

See U.S.P. 1,297,724 of 1919; J., 1919, 363 A.

*Alumina and its salts; Process for the extraction of — from clay.* H. G. Wildman. E.P. 161,310, 14.1.20.

See U.S.P. 1,326,384 of 1919; J., 1920, 191 A.

*Cyanogen compounds; Process for recovering —.* E. V. Espenhahn. Reissue 15,090, 19.4.21, of U.S.P. 1,315,219, 9.9.19. Appl., 17.1.21.

See E.P. 109,254 of 1917; J., 1918, 570 A.

*Zinc sulphide; Manufacture of —.* P. Comment. U.S.P. 1,374,435, 12.4.21. Appl., 23.4.19.

See E.P. 126,274 of 1919; J., 1919, 630 A.

*Mixed acid.* G.P. 303,892. See XXII.

## VIII.—GLASS; CERAMICS.

*Clays; Effect of time on the drying shrinkage of —.* R. F. Geller. J. Amer. Ceram. Soc., 1921, 4, 282–287.

THE drying shrinkages of three shales, two hall-clays, and one fireclay were determined to ascertain how quickly different clays could be dried and to examine the influence of the time of drying on the total shrinkage. The volume shrinkages were determined in each case. It was concluded that the time required for drying depended largely on the water content, but that the structure of the clay was an important factor, fine-grained clays such as hall-clays not being dried at so rapid a rate as clays of coarser grain. The total shrinkage was proportional to the water content and was not influenced by the rapidity of drying.—H. S. H.

*Kaolin; Formation and constitution of —.* V. J. Bernaola. Anal. Soc. Quím. Argentina, 1920, 8, 392–400.

A THEORETICAL discussion of the origin of kaolin from feldspars and the supposed occurrence in it of crystalline particles. (Cf. J.C.S., June.)—G. W. R.

*Silica bricks from quartzite rock; Possibility of preparing high-grade —.* E. Lnx. Stahl u. Eisen, 1921, 41, 258–264.

SILICA bricks can be made from quartzite rock of

a quality corresponding with those made from tertiary quartz. These bricks show under the microscope a high degree of transformation even of the large quartzite grains. Such a transformation is effected in the case of quartzite rock by a high burning temperature such as is employed in America. The degree of transformation of German silica bricks is usually of a much lower order, but microscopical examination of the better bricks shows that by arranging the method of manufacture to suit the raw material, a brick may be made which is not inferior to those prepared from tertiary quartz.—J. W. D.

*Enamel pigment; Manufacture of a red — from cadmium sulphide and selenium.* S. Sugie. Kōgyō-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, 24, 252–258.

RED enamel pigments known as "fire-red" or "cadmium-red" are prepared from a mixture of 80–90% of cadmium sulphide and 20–10% of selenium, the dried and finely powdered components being well mixed and heated in a clay crucible in a muffle furnace at about 700° C. When the blue flame, which burns at the mouth of the crucible, is nearly extinguished, the crucible is removed from the furnace and the product is immediately cooled either by spreading it over a cold place or by pouring cold water over it. The product probably has the empirical formula  $\text{Cd}_2\text{Se}_3$ .—K. K.

*Enamelling oven; An electric vitreous —.* C. W. Mehling and J. W. Carpenter. J. Amer. Ceram. Soc., 1921, 4, 271–276.

AN electric oven eliminates the risk of damaged ware which is always present with oil-, coal-, and gas-fired furnaces and permits the use of the entire heating space. Its initial cost is higher, as is also the power cost per hour compared with that of fuel, but the cost per pound of metal dealt with is actually in favour of the electric oven. Details of the construction are given. The oven, which is wound with nichrome ribbon, can be operated up to 1800° F. (980° C.). The finish of the ware is more satisfactory than with the older types of furnace.—H. S. H.

## PATENTS.

*Electric insulating material; Manufacture of —.* E. F. A. Bültemann. E.P. 137,326, 2.1.20. Conv., 29.1.18.

A FIREPROOF, heat-resisting insulating material, insensitive to moisture, is prepared by mixing substances capable of setting hydraulically, such as cements, plaster, magnesium, oxychloride, etc., with easily fusible insulating substances, such as resins, bitumens, asphalt, sulphur, etc., and vegetable or mineral fibrous filling material, such as cellulose, asbestos, peat, slag wool, glass wool, etc. The mixture is treated with water, preferably under pressure, allowed to harden, dried, and heated, whereby the fusible substances soften and fill up the pores of the mass.—J. S. G. T.

*Refractory insulation.* P. G. Willetts. U.S.P. 1,374,538, 12.4.21. Appl., 17.12.19.

A REFRACTORY heat-insulating material is prepared by burning a mixture of finely divided lignite and plastic fireclay having a high silica content.—J. S. G. T.

*Ceramic pastes; Process and apparatus for de-watering —.* O. Frank. G.P. 330,224, 31.10.18.

IN order to reduce the water content of ceramic pastes so as to leave a mass suitable for shaping, the paste is placed in an apparatus consisting of clay plates with narrow feet so arranged that there is a hollow cavity between them. By reducing the

air pressure in this chamber the excess water is sucked through the top plate and is removed by passing a current of air through the chamber.

—A. R. P.

*Kiln, especially for enamelled ware.* A. Sommer. G.P. 325,747, 15.12.18.

THE kiln is heated indirectly by hot gases conveyed through channels of refractory brick running along the side walls and roof, but at a distance from the same.—L. A. C.

*Refractory and insulating products; Manufacture of —.* G. L. Dimitri and J. E. Delaunay. U.S.P. 1,374,498, 12.4.21. Appl., 21.6.20.

SEE E.P. 142,513 of 1920; J., 1921, 348 A.

## IX.—BUILDING MATERIALS.

*Gypsum; Relation between the fineness and other properties of calcined —.* W. E. Emley and F. C. Welch. J. Amer. Ceram. Soc., 1921, 4, 301—305.

CALCINED gypsum was screened to provide material of different sizes. Each size was tested for normal consistency (measured by the Southard viscosimeter), time of set (measured by a Vicat needle), tensile strength, combined water, and plasticity (measured by spreading a paste of normal consistency upon a porcelain plate of known absorptive capacity at a constant rate). The results indicated that the finer the calcined gypsum the more water was required to make a paste of normal consistency and the more quickly did it set. Within the limits of the specifications of the American Society for Testing Materials, increased fineness of the calcined gypsum was accompanied by increased strength of the set material, but any increase of fineness beyond this resulted in decreased strength. The finer the calcined gypsum the more sand would it carry.

—H. S. H.

*Blast-furnace slags; Experiments on —.* H. Burchartz. Stahl u. Eisen, 1921, 41, 193—200. (Cf. J., 1920, 573 A.)

EXPERIMENTS on the suitability of blast-furnace slag for incorporation in concrete mixings have been extended to include ferroconcrete exposed to the action of sea water. Here the durability of the concrete depends largely on its density, and in this respect the concretes tried proved satisfactory and at least equal to those made with sand. The standard mixture consisted of 1 pt. (by vol.) of cement, 2 pts. of slag dust, and 3 pts. of slag particles of grade ranging from 7 mm. to 40 mm. Compression tests were made on specimens after various periods of hardening up to 3 years. Slag concretes were stronger at all periods than sand concretes. They were also sufficiently impervious to prevent percolation of sea water and rusting of the iron cores.—H. J. H.

*Blast-furnace slags; Strength of —.* H. Burchartz. Stahl u. Eisen, 1921, 41, 472—475.

THE view that the suitability of slag as building and road-making material can be correlated with its colour and composition has been examined by experiments on ten samples of blast-furnace slag. The samples were tested in compression, while resistance to wear was measured by noting loss in weight when the specimens were placed in a rotating drum for half an hour. The mean compressive strengths of the samples ranged from 768 to 2414 kg. per sq. cm. High mechanical strength appeared to be associated with dark colour and high content of iron and manganese. Resistance to wear was associated with compressive strength, but the connexion was not quantitatively rigid. The range

of samples was, however, too restricted (seven being from one works) to allow of a wide generalisation.—H. J. H.

## PATENTS.

*Cement; Process of curing —.* G. L. Smith. U.S.P. 1,374,403, 12.4.21. Appl., 24.7.18.

ARTICLES of cement, cement mortar, or cement concrete are heated in air or a gas under a pressure greater than that of saturated steam at the temperature of the moisture within the pores of the material.—L. A. C.

*Fibrous material [e.g., wood]; Method of treating —.* W. Weimar. U.S.P. 1,375,125, 19.4.21. Appl., 5.2.20.

THE material is rendered non-absorbent, and its insulating properties and texture are improved by successive immersion in a hot solution of sodium carbonate, a hot hydrocarbon liquid, and a solution of sulphur and resin at a temperature sufficiently high to keep the sulphur molten; the material is dried after the first two immersions, and after it has remained in the third solution for a sufficient period to fill the pores it is suddenly cooled.

—L. A. C.

*Heat insulators; Manufacture of moulded —.* P. Krause. G.P. 329,409, 29.3.19. Addn. to 317,170.

MOULDED articles prepared as described in the chief patent from the waste sludge of iron oxide and silicates obtained in the manufacture of alumina are heated, with or without the addition of a flux or an alkali, until they are partially vitrified. The porosity of the articles may be increased by adding combustible material, such as sawdust or cork waste, to the mixture before heating.—L. A. C.

*Impervious acid-resisting apparatus; Manufacture of — from cement.* Freskoschmelz- und Mosaikwerke G.m.b.H. G.P. 332,940, 29.7.17.

A MIXTURE of cement with quartz or similar material and a flux is burnt, and the resulting mass, after grinding, is mixed with a further quantity of the flux and a binder, e.g., Portland, Sorel, or zinc cements, water-glass, or a mixture of water-glass and an alkaline-earth oxide. The mixture is made into the required shape, which is then glazed and fired.—A. R. P.

*Cement; Manufacture of —.* K. G. Wennerström. E.P. 143,243, 12.5.20. Conv., 10.2.19.

SEE U.S.P. 1,343,948 of 1920; J., 1920, 573 A.

*Impregnated wood.* G.P. 334,307. See IIa.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*[Steel] process; Deoxidation processes in the basic Bessemer —.* O. von Keil. Stahl u. Eisen, 1921, 41, 605—611.

A CAREFUL examination was made of two charges at each of nine steelworks. The oxygen was estimated by reduction at 950° C. An increase of oxygen was noticeable during the after-blow in two cases only; in other cases there was a diminution. The average limiting values of oxygen before deoxidation were 0.075 and 0.095%, and after deoxidation 0.030 and 0.040%. Percentages of oxygen determined by reduction at 950° and 1170° C. did not indicate any connexion between the progress of the smelting or the method of deoxidation and the percentage of oxygen. It was found that with less than 0.4% of manganese the oxygen value was high,

and with over 0.5% the oxygen value was lower. On account of the uncertainty of determining the oxygen completely by the method used, co-ordination between the oxygen percentages and the stages of the process cannot be affirmed. Further work on synthetic charges of known oxygen content is also necessary to establish the exact influence of manganese.—J. W. D.

*Basic slag; Influence of basicity of — on the working results of the converter.* L. Blum. Stahl u. Eisen, 1921, 41, 69–74.

THE basicity of basic slag is limited by the proportion of free lime remaining in excess of that required for the formation of tetracalcium phosphate and calcium monosilicate. From consideration of a large number of works analyses it is concluded that for an economical basic process in regard to efficient dephosphorisation and low manganese consumption as well as to effective high desulphurisation and low loss of iron, the excess lime calculated as above should be about 4%.—J. W. D.

*Iron and steel; Roentgen spectrographic investigations of —.* A. Westgren. Iron and Steel Inst., May, 1921. [Advance proof.] 23 pages.

THE results of the investigations show that  $\alpha$ -iron has a cube-centred cubic lattice structure, thus verifying Hull's result (Phys. Rev., 1917, 9, 84; 10, 661). At 800°–830° C., within the so-called  $\beta$ -iron interval, the iron atoms are oriented similar to those of  $\alpha$ -iron. Adopting the view that allotropy is synonymous with polymorphy for solid crystalline substances, this means that  $\beta$ -iron cannot be considered as a separate modification of iron. In austenite and in iron at 1000° C. the crystals have face-centred cubic lattices. This is a characteristic of  $\gamma$ -iron and a fundamental crystallographic difference between  $\alpha$ - and  $\gamma$ -iron. In martensite iron is present as its  $\alpha$ -modification. This is also the case in high-speed steel of ordinary composition hardened at 1275° C.—J. W. D.

*Iron; "Slip-lines" and twinning in electro-deposited —.* W. E. Hughes. Iron and Steel Inst., May, 1921. [Advance proof.] 9 pages.

ETCHED sections of electro-deposited iron, deposited under various conditions, exhibited peculiar lines or markings that appeared sometimes, and especially at low magnifications, as wave-lines running through the mass of the metal, and sometimes, on individual grains, in herring-bone arrangement. It is suggested that these lines are "slip-lines" which are produced in the grains of which the deposit is composed by the forces of contraction that act during the deposition.—J. W. D.

*Iron; Solid solution of oxygen in —.* J. E. Stead. Iron and Steel Inst., May, 1921. [Advance proof.] 5 pages.

WHEN iron is heated in air or oxidising gases, the surface layers apparently absorb oxygen which passes into solid solution, and when the solution becomes supersaturated the oxide separates forming globules of free oxide. These globules become larger and larger as oxidation proceeds, eventually joining together to form continuous layers. Oxidising gases find their most facile passage into the steel between the crystals. The gradual changes from solid solution to free oxide can be clearly traced along the oxidised junctions. Sulphur in the gases, possibly as sulphur dioxide, penetrates into the body of the crystals, producing manganese sulphide, and possibly protoxide, which appear in globular form.—J. W. D.

*Iron; Cupric etching effects produced by phosphorus and oxygen in —.* J. H. Whiteley. Iron and Steel Inst., May, 1921. [Advance proof.] 13 pages.

DIFFERENCES in the phosphorus content of less than 0.02% in adjacent parts of otherwise pure iron can be readily discovered by means of cupric etching reagents. As the difference increases up to 0.15% the contrast becomes more and more pronounced. These differences were produced by first phosphorising strips of iron with phosphorus in hydrogen at 1000° C. and then welding, in hydrogen or other gas, a pile made up of alternate strips of phosphorised and unphosphorised material. Attempts to produce in a similar way an unequal distribution of oxygen which could be detected by cupric reagents failed. When oxygen was present white resist-lines were formed only at the weld junctions, but these lines were not formed where unoxidised iron was welded in dry hydrogen. Two methods of obtaining an unequal oxygen content were used: first by soaking pure iron in its own oxide at a high temperature and then welding it with the unoxidised material; second by reducing highly oxidised iron in hydrogen and then welding it with the original metal at a low temperature.—J. W. D.

*Iron; Baumann sulphur test and the behaviour of phosphorus in —.* P. Oberhoffer and A. Knipping. Stahl u. Eisen, 1921, 41, 253–258.

INVESTIGATIONS were carried out on iron containing from 0.045 to 0.139% of sulphur, iron containing from 0.67 to 1.33% of phosphorus, and iron containing small percentages of both phosphorus and sulphur. As regards the Baumann sulphur printing test, results indicate its usefulness under all circumstances. A side reaction which occurs in the alloys containing phosphorus, due to the formation of hydrogen phosphide, is so small that it does not affect the bromide paper to any marked extent. With pure iron-phosphorus alloys annealing temperatures from 1200° C. upwards and a short heating at 1300° C. remove the dendritic phosphorus structure. This is the case only with pure iron-phosphorus alloys since the presence of carbon or other elements in solid solution in technical irons, such as steel, renders the homogenising so difficult that up to the present no practical heat treatment has been found sufficient to remove the dendritic segregation.—J. W. D.

*Sulphur in steel; Comparison of different methods of estimating —.* T. E. Rooney. Iron and Steel Inst., May, 1921. [Advance proof.] 13 pages.

IN the great majority of cases drillings of ordinary commercial carbon steels need not be annealed before estimating sulphur by the evolution method unless incipient rusting has taken place or the presence of elements such as titanium is suspected. Annealing may be necessary with steel low in manganese and containing chromium and liable to contain oxides which may form an eutectic with the sulphide. No definite conclusions could be drawn from experiments carried out on nickel-chromium steels, including annealing in hydrogen, as only two of the samples were of the type yielding low results by the evolution method, and further work is necessary to determine the cause of this condition in nickel-chromium steels.—T. H. Bu.

*Pig iron containing manganese; Production of — from low-manganese residues, especially Siegerland blast-furnace slag.* H. Thaler. Stahl u. Eisen, 1921, 41, 249–253, 338–343.

A SERIES of experiments was carried out with Siegerland blast-furnace slag containing approximately 12.25% MnO. Different charges of this slag

mixed with varying proportions of iron ores were reduced with carbon using calcium phosphate and limestone as a flux. The reductions were carried out in a small electric furnace, and pig irons containing manganese and ferro-manganese alloys were produced. Economical results were obtained both as regards the percentages of manganese reduced and also as regards cost.—J. W. D.

*Quenching cracks [in steel]; Cause of —.* K. Honda, T. Matsushita, and S. Idei. *Iron and Steel Inst.*, May, 1921. [Advance proof.] 16 pages. (*Cf. J.*, 1919, 582 A.)

OWING to the presence of arrested austenite in martensite the periphery of quenched small pieces of steel is harder than, as hard as, or softer than the interior according as the quenching is soft, medium, or hard respectively. When the interior is much harder than the periphery, quenching cracks occur, due to the smaller specific volume of the austenite as compared with the martensite of the central portion, which exerts a tangential stress on the periphery. This difference in specific volumes increases as the temperature falls, and cracking usually takes place at room temperature. In the case of a large specimen cracking may take place either at room temperature or in the A1 range. In the latter case the stress is caused by the structural difference between the inner pearlitic and the outer austenitic structures. In a hard quenching, owing to a gradual transformation of the arrested austenite the hardness generally increases with lapse of time.

—T. H. Bu.

*Tungsten tool steel; Prevention of hardening cracks and the effect of controlling recalescence in a —.* S. N. Brayshaw. *Iron and Steel Inst.*, May, 1921. [Advance proof.] 86 pages.

THE changes Ac 1.23 and Ar 3.21 in a tool steel containing 1.1% C and 0.8–0.9% W take place in stages which may be separated from each other if sufficient time be allowed for the process. By annealing bars at temperatures corresponding to the completion of the stages of the change point and afterwards subjecting them to a standard hardening process, they were found to lengthen or shorten and respond under bending stress according to the heat treatment received. Milling cutter blanks were heat-treated along with the bars and the blanks machined to a design extremely difficult to harden. The cutters were then subjected to the standard hardening process and it was found that the resulting cracks occur in sequence with the treatment and with the results from the bars. Liability to crack in hardening was greatly reduced when sufficient time was allowed for the heat treatment. Within a certain range of temperature cutters which broke badly in hardening were generally made from blanks in which the ratio of the Brinell hardness number to the Shore figure was comparatively high. It was found that specific heat treatments can be given which ensure a minimum change in length after hardening. The modulus of elasticity of the hardened bars, which differed greatly in other respects, remained remarkably uniform. A standard hardening test is suggested which would give a recognised standard of hardening behaviour.

—T. H. Bu.

*Tungsten steels; Use of very slow cooling in the micrographic study of alloys and the structure of —.* A. Portevin. *Comptes rend.*, 1921, 172, 964–967.

By very slow cooling it is possible to get an enlargement of the whole structural design in alloys in chemical equilibrium, thereby facilitating their micrographical examination; to realise a state of equilibrium or a state nearer to this latter in the case of alloys showing constituents of temper-

ing; to obtain evidence of states not in equilibrium; which have hitherto only been indicated by new equilibrium structures. By the application of this method to tungsten steels containing 0.1–0.4% C and 5–7.5% W, after a reheating to 1300° C. and cooling to 200° C. in 75 hrs. it is possible to obtain a structure with four constituents comprising a ferrite with tungsten, a tungsten carbide, a troostite with tungsten, and a new constituent, a complex of ferrite+Fe,W.—W. G.

*Rusting experiments with sheet iron containing copper.* O. Bauer. *Stahl u. Eisen*, 1921, 41, 37–45, 76–83.

TWO series of experiments were carried out with iron sheets containing increasing percentages (0.09–0.48) of copper. In the first series large sheets were used with the annealing or rolling skin unbroken. The influence of the copper is only plainly marked in industrial districts where iron high in copper corrodes less readily than iron poor in copper. In pure air or in sea air slight additions of copper have in general little effect. This also applies to rusting tests carried out in sea water and on buried material. In the second series of experiments the sheets were small and were without an annealing or rolling skin. With sheets of this nature the results obtained in pure air were not distinct nor reliable, and in distilled water, tap water, or North Sea water the varying percentages of copper were without effect. In water containing much carbon dioxide in solution copper slightly reduced the rusting. Small copper additions also protected against attack by sulphuric acid, and partly counteracted the influence of high percentages of phosphorus.—J. W. D.

*Iron and steel; Corrosion of —.* T. S. Fuller. *Trans. Amer. Electrochem. Soc.*, 1921, 337–346. [Advance copy.]

RELATIVE rates of corrosion of iron were determined by observing the formation of rust caused by a single drop of water placed on a roughly polished surface of the metal. Results of oxidation are perceptible in 1 min. and well marked in 15 mins. By applying successive drops on the same spot, after complete evaporation of a previous drop, the initial rate of corrosion was shown to be much greater than that of later action. Zinc inhibited corrosion in its immediate vicinity, but the zinc itself was attacked. Small quantities of aluminium and calcium did not change the initial rate of corrosion, but chromium produced a marked effect. Copper steels proved to be more resistant to corrosion than cold-rolled wrought iron.—C. A. K.

*Iron; Protection of — with paint against atmospheric corrosion.* J. N. Friend. *Iron and Steel Inst.*, May, 1921. [Advance proof.] 4 pages.

A CONTINUATION of previous work (*J.*, 1918, 736 A). Plates of open-hearth steel, cold-rolled, charcoal annealed, and eventually polished, were painted, left exposed to the air for 5 years, cleaned, and the loss in weight determined. Ordinary boiled oils do not offer so good a protection as litho oils owing to the catalyst (drier) accelerating disintegration of the paint film. The best results are obtained by giving the iron first a coat of low pigment content followed by a coat of high pigment and low oil content. The first film then clings tenaciously to the surface of the metal, while the outer coat protects the inner owing to its lower permeability by moisture and greater resistance to mechanical erosion.

—A. R. P.

*Brasses containing tin; Quenching of —.* L. Guillet. *Comptes rend.*, 1921, 172, 1038–1041.

THE mechanical properties of brasses containing tin are considerably improved when the percentage of

tin is sufficiently high for the formation of the special constituent, similar to the  $\delta$ -constituent of bronzes (*cf.* J., 1906, 813), if the conditions of quenching are such that this constituent goes into solution. Thermal anomalies appear with this special constituent, which shows the same transformation as the  $\delta$ -constituent of bronzes. This constituent dissolves at 350° C. in the  $\beta$ -constituent of brass. Quenching at 600° C. considerably improves alloys containing this constituent, there being a marked increase in their breaking strain, resilience, and hardness.—W. G.

**Arsenic; Removal of — from zinc electrolyte by means of hydrogen sulphide.** H. R. Hanley. *Chem. and Met. Eng.*, 1921, 24, 693—696.

A PLANT for the safe generation on a technical scale of hydrogen sulphide and its employment for the precipitation of arsenic from a solution of arsenical zinc fume in sulphuric acid is described, consisting of a rotating horizontal cylindrical generator, charged with a low-grade copper matte and about 75% (of the weight of matte) of sulphuric acid in 20% dilution, and connected through its end trunnions with the precipitating tank by pipes forming a completely closed circulatory system through which the gases are propelled by means of Roots blowers, the attenuated gas after passing through the arsenical solution being returned to the generator. The gas is delivered to the precipitating tank through a perforated coil situated midway up the tank, whilst a portion of the attenuated gas is injected into the bottom of the tank by a second blower in order to keep the liquid in agitation, and prevent the precipitation of zinc sulphide by a local excess of hydrogen sulphide. The last traces of arsenic are removed by precipitation as an iron arsenic compound, as complete removal by hydrogen sulphide would cause co-precipitation of zinc. The arsenic solution is given a preliminary treatment with sulphur dioxide to reduce arsenates if present, before the hydrogen sulphide treatment.—G. F. M.

**Sintering process; Investigation of the —.** K. Endell. *Metall u. Erz*, 1921, 18, 163—177.

IN the making of agglomerates for smelting from finely divided ores, flue dust, and other residues, the aim should be to produce a body which is porous and mechanically strong at 1000° C., has the greatest possible surface, and requires the minimum of fuel in the subsequent smelting process. Sintering of the mass is usually brought about either by an increase in the grain size of one or more of the constituents with consequent intercrystallisation of the particles, in most cases accompanied by an increase in the specific gravity, or by reactions between the solid constituents of the mass resulting in an evolution of heat and partial fusion of the particles, or in the formation throughout the mass of interlocked crystals of a new compound. The course of the changes that take place during sintering may be followed by the construction of a heating curve and by direct observation through a microscope in conjunction with a small electric heating furnace fitted with a thermo-couple. Such an instrument is described in the paper in detail, together with some results obtained by its use.—A. R. P.

**Blast-furnace and cupola slags. Their composition and graphic methods for determining their constitution.** J. E. Fletcher. *Iron and Steel Inst.*, May, 1921. [Advance proof.] 20 pages.

THE author's investigations support the view that blast-furnace slags consist of a solvent silicate of  $2\text{RO}\cdot\text{SiO}_2$  type in which the remaining constituents are dissolved. A clear view of the theory is provided by means of graphic methods. Alumina lowers the fusion temperature, affects the viscosity

of the silicate in which it is dissolved, and behaves as a neutral constituent. The free silica is the active agent in silicifying the hearth metal. The metasilicates when superheated above their melting point become orthosilicates in character, the amount of superheating needed being reduced in the presence of alumina and ferric oxide. The composition of the primary slags is of fundamental importance. Though the mineralogical character of cold slags affords useful information, it is necessary to ascertain the most probable composition of the liquid slags at the time the reactions are in progress. As the alumina content in blast-furnace slags increases, the silica activity decreases. As the lime content of cupola slags increases, their character approaches that of blast-furnace slags.—T. H. Bu.

**Blast-furnace gas; Cleaning of —.** S. H. Fowles. *Iron and Steel Inst.*, May, 1921. [Advance proof.] 21 pages.

THE use of blast-furnace gas for boiler firing shows only 20% efficiency as compared with its use in large gas engines, and the problem of the most efficient use of blast-furnace gas (*i.e.*, in internal combustion engines) rests largely on a satisfactory gas cleaning process. Internal wet cooling of the gas is condemned because of the loss of potash, and because the dew point of the gas is raised, necessitating a higher degree of preheating of the gas before entering the hog chamber to avoid deposition of moisture. In a plant erected by Palmers' Shipbuilding and Iron Co. the crude gas passes through dry coolers through which pass four vertical annular tubes. Water sprays in the inner tubes effected a temperature fall of 40°—45° C., but good results are obtained by forced air cooling. The gas then passes through the preheaters and filter-bags, the whole of this system being under suction, as more rapid cooling and dust separation were effected under this condition than when the cleaning plant was under pressure. The temperature at the filter-bags should not be below the dew point of the gas. The ideal condition to be aimed at is to cool the gas externally, before cleaning, to a point approaching a dew point of 15°—25° C., as the cleaned gas would then be directly available for use in a gas engine. The electrostatic type of cleaner allows the sensible heat of the gas to be retained, but the gas is not sufficiently freed from dust for use in gas engines and the power costs are high. Operating details of the plant are given in an appendix.—C. A. K.

**Corrosion of boiler tubes.** Worth. See I.

**Blast-furnace slags.** Burchartz. See IX.

**Copper.** Doughty and Freeman. See XXIII.

#### PATENTS.

**Furnaces [;Reverberatory —].** J. L. Gal. E.P. 122,395, 19.9.18. *Conv.*, 12.1.18.

THE roof of a reverberatory furnace is of parabolic or other suitable geometrical form and arranged so that the material to be heated receives the whole of the reflected heat rays from the fire-hearth or other source of heat. In contact with, or spaced from, the interior of the crown is a covering of trellis work coated with material of high absorptive and emissive power such as cerium oxide, and the exterior may be similarly covered with material of low emissive power, such as copper sheet. The trellis work may be heated directly by Bunsen burners.—B. M. V.

**Furnaces; Heating device for — particularly applicable to furnaces for the melting of steel and other metals.** A. Festa. E.P. 147,190, 7.7.20. *Conv.*, 7.12.18.

IN a reversible furnace fired with liquid fuel, the fuel is ignited (and partially distilled) on trays in a

chamber at the end of the furnace. Air required for the combustion of the fuel, after passing through a hot regenerator, is conveyed through a fine close to the chamber containing the fuel trays, and meets the products of partial combustion after entering the furnace chamber.—C. A. K.

*Solder for aluminium and other metals.* P. A. Agostini. E.P. 156,665, 6.1.21. Conv., 5.1.20.

A SOLDER particularly suitable for soldering aluminium to copper or to another piece of aluminium contains at least 50% of zinc, together with tin, copper, and aluminium, preferably in the proportions, 30–35% Sn, 1–2% Cu, 12–15% Al, and 50–53% Zn.—A. R. P.

*Pre-leaching apparatus for use in the treatment of calcined ore and the like.* E. C. Vigeon and J. McConway. E.P. 161,375, 6.2.20.

DAY ore or the like on its way from the roaster is sprinkled with a liquid while in a conveyor, preferably of the screw type.—B. M. V.

*Tin scrap; Method of obtaining iron and tin from —.* P. A. Mackay. E.P. 161,654, 9.1.20.

TIN is removed from tin scrap, leaving bright iron, by immersing the scrap in diluted oleum, the concentration of free sulphur trioxide being maintained at about 10%. The temperature is preferably kept at 45°–50° C.—C. A. K.

*Gold-separator; Electrochemical —.* H. P. Ewell. U.S.P. 1,374,370, 12.4.21. Appl., 7.11.18.

THE apparatus consists of an amalgamating chamber into which the ore is continually fed by mechanical means and a second chamber in which liquid sodium amalgam is formed, and from which it is fed continuously into the amalgamating chamber. The whole charge passes from the latter to a shaking pan, where the amalgam is drained off from the tailings and passed to a device where it is treated to remove the excess of mercury from the gold amalgam. The recovered mercury is returned to the chamber in which the sodium amalgam is made.—A. R. P.

*Ore; Process of treating —.* C. G. Collins, Assr. to C. A. Stevens. U.S.P. 1,374,434, 12.4.21. Appl., 13.1.20.

ORE is heated to expel, and treated so as to prevent the re-entry of, air and moisture. The ore is then concentrated.—C. A. K.

*Treating liquids with gases; Apparatus for —.* Flotation apparatus. W. E. Greenawalt. U.S.P. (a) 1,374,445, (b) 1,374,446, (c) 1,374,447, (d) 1,374,499, and (e) 1,374,500, 12.4.21. Appl., (a) 4.11.19, (b) 16.10.19, (c) 26.5.16, (d) 27.12.15, and (e) 30.10.15. Renewed (a) 25.5.20, (c) 22.3.20, (e) 12.2.20.

(A) A TANK is fitted with a rotary atomiser mounted on a vertical shaft. Liquid in the tank is caused to flow to the centre of the atomiser, and the gas and liquid are mixed and ejected together through outlet holes arranged radially in the atomiser. (B) Gas is passed through a hollow shaft and rotary atomiser into the liquid in the tank. (C) A vertical shaft suspended in a tank is provided with a number of perforated discs which rotate between similar fixed discs. Gas is introduced into the bottom of the tank and is atomised by rising through the series of discs. (D) A hollow member on a vertical shaft rotates in close proximity to stationary baffles, and an electric current is passed from the rotating to the fixed parts. (E) Gas may be passed through a hollow shaft and into a hollow rotating member under the liquid. The drum is provided with radial outlets for the gas.—C. A. K.

*Electrolytic process [for copper deposition].* F. S. Woodward. U.S.P. 1,374,541, 12.4.21. Appl., 25.7.19.

COPPER is deposited from an acid bath, using an insoluble anode, and in restoring the bath to normal strength the resulting increase of acidity only is neutralised.—J. S. G. T.

*Pickling process.* J. Coulson, Assr. to Westinghouse Electric and Mfg. Co. U.S.P. 1,374,552, 12.4.21. Appl., 8.10.17.

A FERROUS metal is pickled by subjecting it to the action of an electric current in the presence of an electrolyte containing sulphuric acid, the polarity of the metal being reversed during the process.—A. G.

*Electromagnetic separator.* Maschinenbauanstalt Humboldt. G.P. 330,641, 7.8.19.

THE separator consists of two magnets one above the other, with a magnetic roller in between. The lower magnet, between which and the roller the material to be separated is passed, is provided with one or more openings beneath the middle of the roller, through which the non-magnetic material falls, while the magnetic part is carried over the far end of the lower magnet by the action of the roller.—A. R. P.

*Zinc and tin; Process for electro-plating metals with —.* Deutsche Metallveredlungs-G.m.b.H. G.P. 305,583, 22.4.17.

BY depositing zinc or tin from baths of 4°–5° B. (sp. gr. 1.03–1.04) with a low current density (not greater than 2.5 amps. per sq. dm. of cathode area), very thin, strongly adherent, firm coatings of metal may be obtained.—A. R. P.

*Alloys containing graphite; Manufacture of —.* G. H. Wichmann. G.P. 332,914, 16.5.20.

THE granulated alloy is given a coating of graphite and then cast or pressed into the required shape. The graphite remains evenly distributed throughout the metal, which is suitable for use in stuffing-boxes or bearings.—A. R. P.

*Precious metals; Recovery of — from sea-water.* Siemens und Halske A.-G. G.P. 333,758, 17.8.18.

SEA-WATER is passed through absorption filters, preferably of fine-grained charcoal, which are cathodically polarised by the use of small currents of a few milliamperes.—A. R. P.

*Steel.* P. MacGregor and A. Balfour. U.S.P. 1,375,255, 19.4.21. Appl., 18.9.18.

SEE E.P. 129,330 of 1917; J., 1919, 685 A.

*Metals; Process of separating and refining —.* G. Haglund. U.S.P. 1,375,631, 19.4.21. Appl., 5.12.18.

SEE E.P. 121,591 of 1918; J., 1919, 908 A.

*Blast furnaces, gas generators, and the like; Automatic charging apparatus for —.* Eisenwerk Jagstfeld Ges. E.P. 145,539, 22.6.20. Conv., 22.4.18.

*Ore sintering mechanisms.* R. J. Tugwood. From Dwight and Lloyd Metallurgical Co. E.P. 161,491, 7.8.20.

See also pages (A) 375, *Asbestos cloth* (E.P. 153,558). 388, *Blast-furnace slag* (E.P. 139,172). 389, *Ore flotation* (U.S.P. 1,359,037). 396, *Ferro-manganese* (U.S.P. 1,361,036).



## XI.—ELECTRO-CHEMISTRY.

*Water; Electrolysis of —, and an oxyhydrogen gas element.* E. Baur. *Helv. Chim. Acta*, 1921, 4, 325–333.

With the object of storing seasonal water-power, the author has devised an apparatus by means of which fused aqueous sodium hydroxide is subjected to electrolysis, sheet-iron electrodes being employed. When the electrolysis is interrupted, the cathodes generate hydrogen and undergo corrosion, but this does not occur when the apparatus is at work. The purity of the hydrogen and oxygen formed is about 99% and the current yield over 90%. Only the hydrogen is stored and is used subsequently in conjunction with air in an element in which electric current is produced by the union of the hydrogen and oxygen in contact with fused sodium hydroxide, iron electrodes in the form of grids being employed.

—T. H. P.

See also pages (A) 386, *Marsh electrolytic cells* (Marsh). 390, *Electric enamelling oven* (Mehling and Carpenter). 410, *p-Aminophenol* (McDaniel and others). 416, *Silver cathodes* (Doughty and Freeman); *Gold, copper, palladium, and platinum* (Treadwell).

## PATENTS.

*Gas battery.* P. A. Emanuel. U.S.P. 1,359,881, 23.11.20. Appl., 15.5.20.

In a cell provided with electrodes of coiled iron wire coated with iron oxide and asbestos and separated by an asbestos diaphragm and connected respectively with supplies of hydrogen and oxygen, a jet of air under pressure is directed downwards into the centre of the cell towards a vent and by its injector action removes the water which would otherwise collect in the bottom of the cylindrical cell casing.

*Ferromanganese [anodes]; Casting —.* C. G. Fink, Assr. to Chile Exploration Co. U.S.P. 1,361,036, 7.12.20. Appl., 9.7.19.

FERROMANGANESE (78–82% Mn) can be cast in the form of thin plates, free from checks or cracks, and possessing satisfactory mechanical strength for use as anodes in electrolytic processes, if a small quantity of a scavenger, e.g., 3% of aluminium or magnesium, be added to it.

*Electric furnaces.* M. R. Trembour. E.P. 133,954, 13.10.19. Conv., 24.3.16.

SEE U.S.P. 1,333,950 of 1920; J., 1920, 375 A.

*Electric furnaces.* E. L. Smalley. E.P. 152,686, 21.10.20. Conv., 21.10.19.

SEE U.S.P. 1,346,854 of 1920; J., 1920, 726 A.

*Electric furnace.* C. Soncini. U.S.P. 1,375,615, 19.4.21. Appl., 12.4.20.

SEE E.P. 142,836 of 1920; J., 1921, 225 A.

*Electrically heated melting and other furnaces [; Mounting of tubes and crucibles in —].* The Morgan Crucible Co., Ltd., and C. W. Speirs. E.P. 161,603, 7.10.19.

See also pages (A) 390, *Insulating material* (E.P. 137,326). 395, *Gold separator* (U.S.P. 1,374,370); *Electrolytic process* (U.S.P. 1,374,541); *Pickling process* (U.S.P. 1,374,552); *Precious metals* (G.P. 333,768). 398, *Purifying glycerin* (E.P. 146,835).

## XII.—FATS; OILS; WAXES.

*Kapok seed; Indian — as a source of oil.* Bull. Imp. Inst., 1920, 18, 335–337.

INDIAN kapok (*Bombax malabaricum*) yielded on extraction with petroleum ether 22.3% of a bright yellow oil, which deposited a quantity of "stearin"

on standing, and had  $n_D^{20} = 1.461$ . The yield of oil is higher than that generally obtained from other kapok seed, and the oil appears to be of better quality. The material left after the extraction of the oil contains about 36% of crude proteins.

—J. C. K.

*Oil from Cynara cardunculus.* E. H. Ducloux. *Anal. Soc. Quim. Argentina*, 1920, 8, 410–412.

THE oil extracted from the seeds of the "donkey thistle" (*Cynara cardunculus*), a troublesome weed in South America, is deep yellow in colour, tasteless, and aromatic in odour. It has sp. gr. 0.9213 at 15° C.;  $n_D^{20} = 1.4729$ ;  $n_D^{25} = 1.4712$ ; iodine value, 103.5; saponif. value, 197.4; insoluble fatty acids, 95.6%; acid value, 39.8; solidif. pt. of fatty acids 23° C. Its properties are similar in many respects to those of cottonseed oil.—G. W. R.

*Acetyl value of fatty substances; Determination of the —.* E. André. *Comptes rend.*, 1921, 172, 984–986.

THE saponification value of the material is determined before and after acetylation. These values being S and S', respectively, then the acetyl value equals  $S_1 - S[1 + \lambda S / (1 - \lambda S)]$ , where  $\lambda = 0.75$ . The values obtained by this method are always slightly lower than those obtained by the method of Lewkowitsch (cf. J., 1890, 660; 1897, 503).—W. G.

*Decolorising agents for the fat industries.* J. Zifferer. *Oel- u. Fettind.*, 1920, 2, 63–64, 87–88, 112–113, 134, 160, 183–184. *Chem. Zentr.*, 1921, 92, II., 861–862.

THE only suitable carbons are those made from materials which do not fuse on charring; hence proteins, glue, etc. give the best products when carbonised with potassium carbonate or sodium potassium carbonate. Carbons containing nitrogen are more active than those containing none. In diminishing order of activity the chief carbons are: animal charcoal from ferrocyanide residues, blood charcoal, bone charcoal, glue charcoal, soot, wood charcoal, and sugar charcoal. Nodules of the size of peas should be used for aqueous solutions, but for oils finely ground carbon is best. For the fat industry bone char is of much less importance than charcoal from ferrocyanide manufacture; the latter cannot be revived. Florida fuller's earth (*Floridin*) is active in the air-dry state at 18° C. but more so at 120° C. or when dehydrated at 300°–400° C. The oil is treated with 2–5% and warmed. The earth can be revived by various methods, but it recovers only two-thirds of its original activity. Salad oils decolorised by silicates often have a slight rancid flavour, and for this reason sodium bicarbonate is frequently added. An earthy flavour is eliminated by washing with 10% brine.—J. H. L.

*Oxidised fatty acids in olive oils obtained by extraction with carbon bisulphide; Determination of —.* F. Goldschmidt and G. Weiss. *Seifenfabr.*, 1920, 40, 649. *Chem. Zentr.*, 1921, 92, II., 815–816.

CONTRARY to Stadlinger's results (J., 1920, 663 A), the authors obtained practically the same values for the oxidised fatty acids in three samples of olive oil high in oxidised acids obtained by extraction with carbon bisulphide, when using different samples of petroleum spirit, including one containing 4% of xylol.—A. de W.

*Fats; Influence of light on animal and vegetable —.* H. Much and H. Schmidt. *Z. Immunitätsforsch. Exp. Ther.*, 1920, 31, 169–200. *Chem. Zentr.*, 1921, 92, I., 645.

THE colour of animal and vegetable fats is destroyed by the combined action of light and oxygen; light of short wave-length has the most effect. If the



fats are dissolved in solvents, the effect of light is least in an ethereal solution and greatest in a carbon tetrachloride solution. It is possible that the colour of animal fats is derived from carotin-like substances present in the food of the animals.

—W. P. S.

*C<sub>18</sub> fatty acids. I. Non-identity of elaeostearic acid tetrabromide from tung oil with ordinary linolic acid tetrabromide.* B. H. Nicolet. J. Amer. Chem. Soc., 1921, 43, 938–940.

CONTRARY to the views of Kametaka (J., 1903, 1198), it is shown that the tetrabromide obtained from  $\alpha$ -elaeostearic acid isolated from tung oil is not identical with linolic acid tetrabromide. It is identical with  $\beta$ -elaeostearic acid tetrabromide (Morrell, J., 1912, 1189). (Cf. J.C.S., June.)

—W. G.

*Hydrogenation of oils; New catalyst for* —. G. Grote. Seifensieder-Zeit., 1920, 47, 713–714. Chem. Zentr., 1921, 92, II., 812.

A CATALYST, with which the time of hydrogenation can be reduced to 10–15 mins. and the temperature to 160° C., is prepared by treating an aqueous solution of a nickel salt with a solution of a magnesium or aluminium salt so as to obtain a colloidal precipitate (double silicate) containing the whole of the nickel and magnesium or aluminium. The voluminous gelatinous precipitate is washed, dried, powdered, and heated in a stream of hydrogen, and the dark-grey reduction product stored in oil.—A. de W.

*Beeswax; Detection of very small quantities of — in admixture with mineral oil and colophony.* A. Jolles. Oel- u. Fettind., 1920, 2, 207, 231–232. Chem. Zentr., 1921, 92, II., 862.

COMPARATIVE experiments were made with mixtures of which one contained 70% of colophony and 30% of mineral oil and the other 67% of colophony, 30% of mineral oil, and 3% of yellow beeswax. The saponification values of the original mixtures proved useless for the detection of beeswax. By extraction with cold 70% alcohol, however, the colophony was removed, and the residue in the case of the wax-free mixture gave saponification and acid values of practically 0, whilst in the other case the saponification value was 8.41, the acid value 1.70, and the Hübl iodine value 3.94, the calculated values being 8.46, 1.80, and 3.67 respectively. It was found possible to separate the wax from the mineral oil by means of petroleum ether, in which the former is insoluble.—J. H. L.

*Carnauba wax palm; Utilisation of the* —. C. Grimme. Pharm. Zentralh., 1921, 62, 249–257.

THE S. American carnauba wax palm (*Copernicia cerifera* Mart.) yields an edible fruit containing about 10% of oil, the greater part of which is found in the seeds; the roots of the tree are stated to have certain medicinal properties, the trunk consists of a very hard wood, and the leaves are useful for a variety of purposes (thatching, paper-making, thread making, etc.). The young green leaves are edible, and from them exudes the well-known carnauba wax.—W. P. S.

*Hydrogenated phenols and their use in the soap industry.* W. Schrauth. Seifenfabr., 1921, 41, 129–132. Chem. Zentr., 1921, 92, II., 814.

THE mixture of cyclohexanol and the three isomeric methylcyclohexanols, known under the trade name of "hexalin," is used as a substitute for castor oil products hitherto used in technical soaps. In spite of its small solubility in water and its oily nature, hexalin yields clear permanent solutions with aqueous soaps, even when present in an amount

substantially in excess of the soap. A suitable mixture consists of oleic acid, 500 kg., alkaline lye (50° B., sp. gr. 1.53) 200 kg., hexalin 600 kg., water 400 kg. Special advantages are claimed for hexalin soap mixtures in wool-scouring, chemical cleaning, emulsification, and in the preparation of cutting oils, shoe creams, plant sprays, etc.—A. de W.

*Glycerin; Viscosity of distilled* —. J. Kellner. Z. deuts. Oel- u. Fettind., 1920, 40, 677–678. Chem. Zentr., 1921, 92, II., 834.

THE viscosity of a crude glycerin distillate, with an ash content of 0.001% and sp. gr. 1.24 at 15° C., was reduced by the refining process from 35.5° Engler to only 16.75° at 24° C. The latter value corresponds with an almost chemically pure glycerin, according to tables given in the paper. Viscosity measurement is recommended therefore as a valuable test of the purity of glycerin.—B. V. S.

*Wax-colophony mixtures.* Jahn. See XIII.

*Influence of fats on growth of bacteria.* Much and Schmidt. See XVIII.

#### PATENTS.

*Oils and fats; Process of refining* —. K. Stiansen. E.P. 141,028, 9.3.20. Conv., 21.3.19.

OIL, e.g., whale or herring oil, is agitated at 40°–50° C. with about 5% of its weight of powdered, calcined apatite or other mineral phosphate, and is subsequently allowed to stand at the same temperature, whereby the apatite settles to the bottom together with impurities present in the oil. Solid fats are treated at a temperature sufficiently high to render them fluid.—I. A. C.

*Animal and vegetable oils; Refining and decolorising* —. A. S. Quick. E.P. 161,813, 20.3.20.

To every 20 galls. of the oil an equal quantity of water and 10 lb. of monazite sand or other radioactive material are added, the mixture is heated to a temperature below that at which it is chemically altered, e.g., about 120° F. (49° C.), and stirred continuously for about 6 hrs., allowed to settle, and the oil separated.—A. de W.

*Fatty acids and glycerides thereof; Process of treating [dehydrogenating]* —. H. A. Levey. U.S.P. 1,374,539, 12.4.21. Appl., 19.9.16.

FATTY oils are dehydrogenated by subjecting them to the action of a gas when mixed with catalytic material at a temperature in excess of that at which hydrogenation is effected under the same conditions by passage of hydrogen.—A. de W.

*Sulphated oil.* F. L. Randel. U.S.P. 1,374,607, 12.4.21. Appl., 11.12.19.

A FATTY or oily material is mixed with sulphuric acid *in vacuo*.—A. de W.

*Fats; Apparatus for the extraction of — from moist, granular, or powdery materials.* A. Lorenz. G.P. (A) 333,492 and (B) 333,493, 25.7.17.

(A) The cover of the extraction vessel slopes steeply from one side to the other and is pierced in the centre by the shaft of the agitator. The solvent containing the fats is withdrawn through the highest part of the cover, and the outlet is fixed on one side of the agitator, which is driven from the top. The pulley is attached on the low side of the cover. (B) The closed extraction vessel has a perforated bottom, beneath which the solvent is admitted. At the side of the vessel a box is attached containing a group of valves, arranged one above another, each of them being preceded by a filter. The valves are connected with the outlet, allowing the solvent to be withdrawn from the vessel, after the extraction is completed, without coming in contact with the atmosphere.—C. A. C.

*Glycerin; Process for purifying* —. Elektro-Osmose A.-G. (Gräf Schwerin Ges.). E.P. 146,865, 5.7.20. Conv., 17.1.19.

Crude glycerin is freed from electrically-active impurities, e.g., inorganic salts, fatty acids, soaps, fat, protein, colouring matters, and the like, largely present in the colloidal state, by electro-osmosis, using diaphragms of marked electric potential and carrying a suitable charge to induce passage of the impurities through the diaphragms. Thus, crude glycerin is freed from accompanying acid (negatively charged) impurities by subjecting it, preferably at raised temperature, to an electric current when contained in the cathode chamber of a cell divided into two compartments by a positive diaphragm, such as leather, the acid impurities migrating to the anode chamber which contains water. Similarly, basic impurities migrate to the water contained in the cathode chamber of a cell divided into two compartments by a vegetable (viscose or parchment paper) diaphragm, when the crude glycerin is contained in the anode chamber. The crude glycerin may also be contained in the middle chamber of a three-part cell, the two separating diaphragms of which may be either of negative or of a positive character, or that bounding the cathode chamber of negative or indifferent character, while that bounding the anode chamber is of positive character, the electrodes being in the side cells which contain water.—A. de W.

*Oil; Process for extraction and refining of* —. P. M. Heyerdahl. E.P. 137,514, 6.12.19. Conv., 9.1.19.

See U.S.P. 1,368,148 of 1921; J., 1921, 227 A.

*Soaps; Deodorisation of* —. C. Stiepel. E.P. 145,502, 21.6.20. Conv., 10.8.16.

See G.P. 305,702 of 1916; J., 1918, 476 A.

*Yeast*. E.P. 160,496. See XVIII.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Smalt; Constitution of* —. A. Duboin. Comptes rend., 1921, 172, 972—974.

Using the method previously described (*cf.* Comptes rend., 1892, 114, 1361) the author has produced two blue cobalt compounds of the type of smalt. One has the constitution  $K_2O, CoO, 3SiO_2$ , and the other  $KCl, K_2O, CoO, 4SiO_2$ .—W. G.

*Turpentine; Composition of French* —. M. Vèzes. Comptes rend., 1921, 172, 977—980.

The results obtained by the polarimetric method of Darmon (Thesis, Paris, 1911) for the estimation of the relative amounts of pinene and nopinene present in French turpentines can only be considered as first approximations. Further work is necessary to elucidate the discrepancies found. —W. G.

*Turpentine oil; Determination of petroleum spirit in* —. J. Marcusson. Chem.-Zeit., 1921, 45, 418.

REPLYING to recent criticism by Salvaterra (J., 1921, 186 A), the author maintains the accuracy of a method described previously by himself (J., 1912, 444) and depending on the conversion of the turpentine oil into soluble oxidation products by means of fuming nitric acid.—W. P. S.

*Phenols of low-temperature coal tar* [; *Resins from* —]. W. Glud and P. K. Breuer. Ges. Abhandl. Kennt. Kohle, 1919, 4, 221—236. Chem. Zentr., 1921, 92, II., 855—856.

An investigation was carried out to determine what proportion of the total phenols in low-temperature coal tar could be utilised in the production of "bakelite" and "novolac" by condensation with formaldehyde. The action of the latter in the presence of ammonium chloride as catalyst was tested on the following products: Crude tar from which the most viscous compounds had been removed, the fractions distilling between 150° and 250° C. and between 250° and 300° C., including the hydrocarbons that distil at these temperatures, the cresol fraction, and the phenols obtained from the lubricating oil and resinous fractions. The hydrocarbons present did not interfere with the reactions and could readily be removed from the products, which were solid, brittle resins having the properties of the "novolacs." Condensation of a crude tar-cresol mixture with benzaldehyde and paraldehyde yielded viscous semi-solid products which could be changed into solid resins by heating under pressure. The colour of the resins varied, according to the material from which they were produced, from light yellow to dark brown, and they could be made less brittle by leaving behind in the final product a certain amount of the hydrocarbons originally present in the raw material. Bakelites A (soluble), B (fusible), and C (infusible) were prepared by condensation in the presence of a basic catalyst such as ammonia, but variety A can only be made from the whole of the phenol fraction of the crude tar or from the whole of the crude tar itself. The bakelites are clear solid substances varying in colour from light yellow to very dark brown, and the A variety yields good varnishes in 50% alcoholic solution. Variety C may be made from A by the use of benzylamine as a catalyst, or it may be produced directly from the phenol fraction boiling below 230° C. or from that boiling between 230° and 260° C. if soda is used as a catalyst. —A. R. P.

*Lead compounds of phenols; Use of* — [in paints]. F. Fischer and V. Ehrhardt. Ges. Abhandl. Kennt. Kohle, 1919, 4, 264—279. Chem. Zentr., 1920, 92, II., 854.

LEAD oxide combines with phenol, cresol, *p*-hydroxydiphenyl, and naphthols in benzene solution to give products which are not very satisfactory as paints. If, however, instead of the pure phenols the crude phenol-rich fraction (middle oil) of low-temperature tar or coke-oven tar is used, the product is a useful paint for protecting iron-work from rust. The difference in the two products is ascribed to the action of the resinous substances produced from the easily oxidisable hydrocarbons in the crude oils by the action of the lead oxide. Oxidation of the crude tar oils and distillation with potassium permanganate or nitric acid yields varnish-like substances.—A. R. P.

*Wax and colophony; Supersaturated solid solutions of* —. G. Jahn. Kolloid-Chem. Beih., 1921, 13, 213—232.

MIXTURES of beeswax and colophony can be divided into liquid mixtures, solid solutions, and mixtures in which solid solutions are in equilibrium with pure wax. Mixtures containing up to 75% of colophony can form solid solutions, and on melting and rapidly cooling, supersaturated solutions are formed which are more stable the longer the mixture has been kept in the liquid condition. (*Cf.* J. C. S., June.)—J. F. S.

*Protection of iron*. Friend. See X.

## PATENTS.

*Resin-like bodies; Production of* —. M. Melamid. E.P. (A) 133,712 and (B) 133,713, 11.10.19. Conv., 4.2.18.

(A) RESIN-LIKE substances are obtained by treating aromatic sulphochlorides with salts of resinic, humic, or lignoceric acids; *e.g.*, to a solution of 120 kg. of sodium colophionate in 100–200 kg. of water is added 100 kg. of  $\alpha$ - or  $\beta$ -naphthalene-sulphochloride with stirring, the solution being heated to 30° C. for 12 hrs. The liquid is allowed to cool, and the solid product separated by decantation or filtration, and dissolved for the purpose of purification in an organic solvent, *e.g.*, benzol, which is then evaporated off. (B) The resinsates or the like are replaced by soft coal-tar pitch, anthracene oil distillates obtained in the production of hard pitch therefrom, or the alkali-soluble phenolic portion of soft pitch or anthracene oil, in the presence of alkali; *e.g.*, 100 kg. of soft pitch containing 20% of alkali-soluble matter, is heated with sufficient caustic soda lye to neutralise the acidity, allowed to cool while stirring constantly, and about 24 kg. of *o*- or *p*-toluenesulphochloride (or a commercial mixture of the same) in benzol added. After stirring for 12 hrs., the benzol solution is separated from the aqueous layer, washed with water until neutral, dried over, *e.g.*, calcium chloride, and the solvent distilled off *in vacuo*.—A. de W.

*Anti-corrosive paint or composition.* C. H. Iverson and G. S. Roberts. E.P. 161,201, 12.7.19.

To a solution of asphaltum in a volatile solvent, *e.g.*, 6 lb. to 0.75 gall. of benzol or petrol, is added 2–5% of a solution of rubber in benzol or other solvent, of a strength of, *e.g.*, 2 oz. to 40 oz. of solvent; preferably a similar amount of a solution of 4 oz. of gum elemi in 2–6 oz. of solvent is also added. The mixture is treated with 1–4% of sulphur, sulphur monochloride, or sulphides such as antimony sulphide, and vulcanised by heating to about 95° F. (35° C.) for about 30 mins. The composition, when cooled, is ready for use, or suitable filling agents such as silica, asbestos, graphite or the like may be added. Metallic stearates, palmitates, oleates, or resinsates dissolved in a volatile solvent may be employed in place of the solution of gum elemi. Alternatively, 0.5–1% of sulphur or a suitable sulphide may be added direct to the molten mixture of asphaltum and rubber, the vulcanised composition being then dissolved in a hot solvent.—A. de W.

*Pigments; Process of making* —. L. R. Baker. E.P. 161,280, 6.1.20.

A solution of commercial zinc sulphate is heated by live steam and blown with compressed air, during which time about 1% by wt. of an alkaline-earth peroxide, *e.g.*, calcium or barium peroxide, and powdered zinc or lead oxide are added. After filter-pressing to remove precipitated iron oxide, the filtrate containing hydrated zinc oxide in suspension is again treated with steam and compressed air, and subjected to another filter-pressing operation. The filtrate is then mixed with a solution of barium sulphide in equivalent amount, the resulting precipitate being filter-pressed, washed, dried, calcined, etc.—A. de W.

*Strontium and barium sulphates; Method of obtaining* — from native sulphates. P. A. Mackay. E.P. 161,655, 9.1.20.

NATIVE strontium or barium sulphate ground to pass a 200-mesh sieve is dissolved in oleum and reprecipitated by dilution. The resulting *blanc-fixe* is superior in colour and fineness to that prepared by grinding and levigation.—C. I.

*Coating composition and process of producing same.* W. Alexander and J. E. Clegg. U.S.P. 1,358,914, 16.11.20. Appl., 25.9.17.

A LIQUID coating composition is composed of a cellulose ester (pyroxylin, celluloid) and a boron compound (dehydrated boric acid) dissolved in a common solvent, *e.g.*, methyl ethyl ketone, which is saturated with the boron compound.

*Finely-divided material [carbon black]; Process of cleansing* —. J. C. Morrell. U.S.P. 1,359,091, 16.11.20. Appl., 22.3.19.

VOLATILE impurities, *e.g.*, adsorbed hydrocarbon oils, are removed from carbon black by heating it to 450°–600° C. in an iron crucible having a perforated false bottom and treating it with superheated steam introduced beneath the false bottom. The process is applicable to the purification of other finely divided materials, *e.g.*, kieselguhr.

*Phenolic condensation product; Process of producing a* —. L. V. Redman, A. J. Weith, and F. P. Brock, Assrs. to Redmanol Chemical Products Co. U.S.P. 1,374,526, 12.4.21. Appl., 16.9.18.

EQUAL parts of 40% formaldehyde and phenol are boiled together in the absence of a catalyst until a viscous gummy lower layer and a supernatant aqueous layer containing approximately 10% of formaldehyde are formed. The aqueous layer is discarded and the mass shaped and subjected to a prolonged heat treatment below 100° C. until it becomes hard, substantially insoluble, and anhydrous throughout. The mass is then subjected to further heat treatment at a temperature exceeding 100° C. for a relatively short period.—A. de W.

*Pitch, colophony, shellac, etc.; Process and apparatus for comminuting materials such as* —. R. Behr. G.P. 333,704, 13.2.18.

A STREAM of the hot liquefied material is conducted over heated distributing plates or discs from which it flows in very fine threads; the latter are cooled and solidified by falling through the air or by passing through an artificially cooled space.—J. H. I.

*Siccatives and binding agents for lacquers and paints.* P. Friesenhahn. G.P. 334,761, 25.7.19.

METALLIC resinsates or linoleates are dissolved in hydrogenised phenols, such as the cyclohexanols or their esters, or in cyclohexanone and its homologues, with or without the aid of other solvents. Solutions of resinsates of aluminium, tin, zinc, manganese, and cobalt in cyclohexanyl formate constitute excellent siccatives. The solutions, with or without linseed oil or linseed oil varnish, can be used as vehicles in paints.—J. H. I.

*Soot-carbon.* E.P. 137,065. See IIA.

*Lakes.* E.P. 143,242. See VI.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Hevea rubber; Use of brackish water in the preparation of* —. W. Spoon. Comm. Central Rubber Stat., Buitenzorg, 1921, No. 25, 1–15.

ON account of the more thorough washing to which it has been subjected, crêpe rubber is less affected than sheet rubber by the use of brackish water, *e.g.*, tidal river water, for the dilution of the latex. The presence of 12% of sea water in the brackish water is sufficient to produce an appreciable effect on sheet rubber, whereas in the production of crêpe rubber the limiting proportion is about 23%.

—D. F. T.

*Rubber; Changes in plantation — after storage.* O. de Vries. Comm. Central Rubber Stat., Buitenzorg, 1921, No. 25, 16—28.

STORAGE for 2—4 years in cupboards does not adversely affect the quality of first-grade rubber, and the tensile strength and slope remain unaltered; the rate of vulcanisation generally shows a small increase, but the viscosity frequently decreases considerably. Lower grades of rubber sometimes undergo marked deterioration on storage. Crêpe from "matured" rubber behaved similarly to first-quality crêpe, except that its rate of vulcanisation decreased. Storage therefore tends to lessen the variability in the rate of vulcanisation of plantation rubber.—D. F. T.

## PATENTS.

*Vulcanisation of rubber.* G. Bruni. E.P. 140,387, 4.3.20. Conv., 15.3.19.

In the presence of a salt of a divalent or trivalent metal with a dithiocarbamic acid derived from a primary or secondary aliphatic amine, e.g., zinc pentamethylenedithiocarbamate, vulcanisation can be effected with as little as from 0.2 to 2% of sulphur.—D. F. T.

*India-rubber products; Manufacture of —.* A. Helbronner. E.P. 142,083, 11.2.20. Conv., 19.4.19.

SURFACE resinification in rubber goods vulcanised in an atmosphere of hot air can be avoided by incorporating a reducing agent, such as quinol, pyrogallol, tannins and derivatives, *p*-aminophenol, formaldehyde derivatives, sulphites, or hydrosulphites. Litharge is commonly used for a similar purpose, but causes the development of a black colour which is avoided with the preceding substances.—D. F. T.

*Rubber products; Manufacture of —.* H. Wade. From Goodyear Tire and Rubber Co. E.P. (A) 161,482 and (B) 161,483, 6.7.20.

(A) AIR-DRIED glue (containing 5—15% of moisture) is introduced into rubber by subjecting a mixture of it with unworked rubber to a milling action in a jacketed container; heat is developed, and the temperature is maintained below 300° F. (about 150° C.) by external cooling. After this operation the cooled mixture needs no further drying, and the remaining compounding ingredients are incorporated in the usual manner. (B) Instead of introducing fine powders, such as carbon black, directly into rubber, the powder is first mixed uniformly into an emulsion of an aqueous jelly or solution of glue with benzene; this mixture is then incorporated into rubber in the usual manner.—D. F. T.

*Rubber, ebonite, vulcanite, and the like substances; Reforming or vulcanisation of —.* H. Gare. E.P. 161,648, 9.1.20.

THE heating necessary for the vulcanisation of rubber or the "reforming" of old vulcanised rubber is effected by a hot circulating solution of which the b.p. is a little above the necessary temperature. A solution of calcium chloride in water is convenient, the proportions for a vulcanising temperature being 3½ lb.:1½ pints (b.p. 270° F., 132° C.), and for reforming, 6lb.:1½ pints (310° F., 154° C.).—D. F. T.

*Rubber; Manufacture of products resembling —.* Badische Anilin und Soda Fabrik. G.P. 329,676, 4.5.18.

THE polymerisation of butadiene and its homologues is considerably expedited by the addition of the polymerisation products obtained in the manner described earlier (F.P. 459,005 of 1913, and Addition thereto; J., 1913, 1164).—D. F. T.

*Rubber; Process of treating —.* J. Porzel, Assr. to Superior Rubber Co. U.S.P. 1,374,231, 12.4.21, Appl., 7.8.18. Renewed 9.10.20.

SEE E.P. 124,887 of 1918; J., 1919, 379 A. Oxygen is excluded during the mixing operation.

*Rubber and the like; Methods of and apparatus for mixing or masticating —.* Farrel Foundry and Machine Co., Asses. of D. R. Bowen and C. F. Schnuck. E.P. 137,820 and 158,279, 9.1.20, Conv., 13.1.19.

## XV.—LEATHER; BONE; HORN; GLUE.

## PATENTS.

*Tanning or impregnating materials; Process for —.* Elektro-Osmose A.-G. (Graf Schwerin Ges.). E.P. 143,921, 28.5.20. Conv., 30.11.18.

AFTER a preliminary tannage or impregnation by the electro-osmotic process described in E.P. 19,849 of 1914 (J., 1915, 1020) the hides or skins are completely tanned or impregnated in pits or casks without the aid of an electric current and preferably in strong liquors.—D. W.

*Catechin and catechutannic acid; Manufacture of —.* E. H. Bray. From Indian Wood Products Co. E.P. 161,431, 24.3.20.

WOOD of the *Acacia catechu*, *A. sendra*, etc., is disintegrated and heated with water, preferably in closed vessels with a steam pressure up to 30 lb. per sq. in. The extract is evaporated, preferably under reduced pressure, to a sp. gr. of 1.05—1.10, when catechin separates on cooling. On further evaporation under reduced pressure the mother liquors yield catechutannic acid.—D. F. T.

*Leather substitute; Process for preparation of a —.* G. Ruth and E. Asser. G.P. 332,666, 13.6.19.

CELLULOSE esters, alone or mixed with esters of naphthenic acids, are incorporated with insoluble metallic naphthenates, such as the aluminium, chromium, iron, and cobalt salts, particularly the aluminium-magnesium salt, and the mixture is treated in the usual way with loading material.—B. V. S.

*Hides; Process for tanning — by means of ferric salts.* Chem. Fabr. vorm. Weiler-ter Meer. G.P. 334,004, 27.2.16.

IN the treatment of hides with solutions of ferric salts the formation of basic salts is avoided by the addition of an acid, such as formic acid, to increase the hydrogen ion concentration, or by employing double ferric ammonium salts, which do not readily hydrolyse, or by adding chromium salts which retard hydrolysis of the iron salts. By this means the hides may be rapidly and uniformly permeated by the iron solution. They are subsequently treated with a neutralising agent, such as sodium carbonate, to increase their durability. Good results are also produced by adding oxidation or decomposition products of glucose or other sugars, such as those obtained by treatment with chromium oxide or chromates, or by addition of vegetable tanning agents, to the solution of iron salts.—W. J. W.

*Colouring of natural horn; Process for making the — water-resisting.* M. Lambeck. E.P. 154,200, 22.11.20. Conv., 26.9.19.

ARTICLES of natural horn are exposed either before or after polishing to the action of formaldehyde, its polymers, or substances capable of yielding formaldehyde.—D. W.

*Mixing or agitating lime in tannery pits; Apparatus for —.* F. J. Boys. E.P. 161,362, 28.1.20.

*Iron salt for tanning.* E.P. 146,214. See VII.

## XVI.—SOILS; FERTILISERS.

*Soils; Amount of unfree water in — at different moisture contents.* G. Bouyoucos. Soil Sci., 1921, 11, 255–259.

FURTHER experimental evidence is given in support of the view that the amount of water which soils are able to render unfree (cf. J., 1921, 231 A) does not vary with the different moisture contents of the soils, but appears to remain constant. This is contrary to the mathematical deductions of Keen (cf. J., 1919, 956 A).—W. G.

*Soil; Lime requirement and reaction of lime materials with —.* C. J. Schollenberger. Soil Sci., 1921, 11, 261–276.

EXPERIMENTS were carried out with a nearly pure limestone of high calcium content, in five degrees of fineness, with calcite, magnesite, and dolomite, ground to pass a 100-mesh sieve, with chemically prepared oxides and carbonates of calcium and magnesium, and with two slags, one a dicalcium silicate and the other a blast-furnace slag. These materials were mixed with soil in undrained pots kept at a suitable moisture content and without a crop. The limestone, calcite, and magnesite were similar in behaviour, approximately one half of an application equivalent to 3½ tons per acre being attacked in 4 weeks and two-thirds in 28 weeks. The dolomite was 50% more resistant than the other materials. Quicklime is more reactive than the precipitated carbonate, although the differences become less marked with time. Chemically prepared magnesium oxide gradually became carbonated, but on the other hand the disappearance of carbonate from an equivalent application of prepared magnesium carbonate was complete within one month. The dicalcium silicate slag reacted with soil as readily as precipitated calcium carbonate, but blast-furnace slag was the least reactive of all the materials tested. Fineness of grinding is of importance, coarsely ground limestone being far less reactive than finely ground limestone. The quantitative relations between bases not residual as carbonate and reductions in lime requirement were found to be reasonably close, a possible consumption of bases by nitrification being considered. Possible sources of error in determining lime requirements, such as manner of preparation of the sample, the temperature, and the time allowed for the determination are considered.—W. G.

*Soil; Comparison of the technique recommended by various authors for quantitative bacteriological analysis of —.* Z. N. Wyant. Soil Sci., 1921, 11, 295–303.

The author recommends the following procedure for the quantitative bacteriological examination of soils. Not less than 10 g. of soil should be used in making the first dilution, which should be not less than 1:10. The weighed soil, 10 g., should be placed in a graduated flask and sufficient of the diluting liquid added to make up the volume to 100 c.c. If the soil contains a considerable amount of organic matter, it should be triturated in a mortar with a little of the diluting liquid. In making further dilutions or plating, an aliquot portion of the soil must be transferred and subsequent dilutions may well be 1:10. Numbers of micro-organisms (total or otherwise) should be estimated on the basis of soil dried to a constant weight at 100° C.—W. G.

*Soils; Analysis of —.* K. A. Vesterberg. Svensk Kem. Tidskr., 1921, 33, 12–22. Chem. Zentr., 1921, 92, I, 752.

THE author questions statements to the effect that salts dissolved in the moisture in soils are absorbed by plants at the same rate as the water itself; the roots do not take up the salts etc. in the proportion in which they occur in the soil. As regards the concentration of plant foods in soil moisture which constitutes the minimum requirement of plants, the following quantities are suggested:— $P_2O_5$ , 1–2; nitrogen, 2–5;  $K_2O$ , 10–20 mg. per litre. Climate would possibly have some effect on the efficiency of these amounts of foods.—W. P. S.

*Soil fumigation experiments with p-dichlorobenzene for the control of the peach-tree borer, Sanninoiden eritosa, Say.* A. Peterson. Soil Sci., 1921, 11, 305–319.

p-DICHLOROBENZENE gave satisfactory results when used as an insecticide for the control of the peach-tree borer. One ounce of the material is applied to the soil in a circular band round the tree and 2 in. from it, on a level with the highest point where gum is exuding from the tree, which is earthed up if necessary. The p-dichlorobenzene is then covered with soil which is packed down. This treatment it is claimed will kill 90–100% of the borers if the soil temperature is 55°–60° F. (13°–15.5° C.) or higher and the soil is not too wet.—W. G.

*[Plant] growth and sap concentration.* H. S. Reed. J. Agric. Res., 1921, 21, 81–98.

OBSERVATIONS on apricot and orange trees show that growth and sap concentration tend to vary in opposite directions. As the season advances there is a gradual increase in sap concentration, this increase being noticeable for some time after active growth has ceased. Of the environmental factors examined, soil moisture was the only one having an obvious effect upon sap concentration, increase in soil moisture causing a diminution in the concentration of the plant sap. Lower concentrations of plant sap in the shoot as a whole appear to be associated with abundant water intake and rapid vegetative growth, higher concentrations with slow growth and fruit-bud formation.—W. G.

*Nitrogen-fixing organism; Influence of uranium salts on a —.* E. Kayser. Comptes rend., 1921, 172, 1133–1134.

THE influence of uranium salts on *Azotobacter agile* depends upon the nature of the salt used, its concentration, and the colour of the containing vessel. Uranium acetate gave an increased fixation of nitrogen and better utilisation of sugar at certain concentrations, but uranium phosphate in every case gave less satisfactory results than the control.—W. G.

*Seeds; Effect of salt solutions having definite osmotic concentration values upon absorption by —.* W. Rudolfs. Soil Sci., 1921, 11, 277–293.

SINGLE salt solutions of calcium nitrate, potassium carbonate, magnesium sulphate, potassium phosphate, sodium chloride, potassium chloride, and sodium nitrate, and one of Shive's three salt solutions  $R_3C_2$  (cf. Shive and Martin, J., 1918, 598 A) were used, the osmotic concentration values of the solutions varying from 0.001 to 7.0 atm. The amounts of absorption from these solutions by different varieties of seeds were determined. The results indicate a marked variation in the absorbing power of seeds of different species, leguminous seeds showing the highest rate of absorption of the seeds examined. The rates of absorption are progressively retarded by increase in osmotic concentration values of the solutions, provided there is

no chemical action on the seed coating, there being an approximately linear relation between these two factors. The retardation of absorption rates is apparently due to osmotic resistance offered to the entrance of water into the seed.—W. G.

*Hydrocyanic acid in plants.* Menaul. See VII.

*Activated sewage sludge.* Fowler. See XIXs.

#### PATENTS.

*Phosphates; Manufacture of assimilable* —. J. J. Morel. E.P. 160,847, 14.8.20.

MINERAL phosphates, consisting of mixtures of tricalcium phosphate and calcium carbonate, are treated in a rotary furnace at 1000° C. with a continuous current of sulphurous gas, air, and steam, the proportions being such as to give a mixture of mono- and dicalcium phosphate and calcium sulphate. The production of these phosphates is facilitated by adding about 1% of a chloride, e.g., calcium chloride, to the phosphatic material.

—W. J. W.

*Organic matter; Treatment of — for fertiliser purposes.* A. J. Grinnell. E.P. 136,829, 16.12.19. Conv., 27.4.17.

See U.S.P. 1,268,563 of 1918; J., 1918, 522 A.

### XVII.—SUGARS; STARCHES; GUMS.

[Sugar] juices which cannot be satisfactorily defecated. O. Muller. Bull. Assoc. Chim. Sucr., 1921, 20, 239—247.

DIFFICULTIES in the defecation of juices from canes damaged by heat or frost are due to the presence of abnormal amounts of acetates and other organic salts which cannot be eliminated. As palliative measures the author recommends carbonation of the juice from the third and fourth crushings, sulphitation of the juice after decantation, and perhaps imbibition with cold water. Cases occur also in which juices from sound canes resist defecation even by large amounts of lime. These are attributed by the author to the presence, in certain canes, of colloidal compounds containing organic and silicious matter not precipitable by lime, which are liberated from the cells ruptured by crushing. Sulphitation is no remedy, but the colloidal matters may be destroyed by superheating the juice at about 116° C. before liming; the organic matter is thereby coagulated and the silica is rendered precipitable by lime. The colour of the juice is also improved, defecation can be effected with about  $\frac{1}{4}$  of the usual amount of lime, and sulphitation is often unnecessary or it may be postponed to a later stage, e.g., that of syrup.—J. H. L.

*Honey; Valuation of artificial* —. V. Stanek and J. Vondrak. Z. Zuckerind. Czecho-Slov., 1921, 45, 203—205.

For the determination of dry substance in artificial honey the authors recommend either the refractometric method, applied to the undiluted material, previously warmed if necessary to dissolve crystals, or the following saccharometric method:—The sp. gr. of a mixture of the sample with an equal weight of water, in degrees Balling, is corrected for temperature if necessary and then multiplied by 2, and from the product 0.7° is subtracted to allow for the effect of contraction on dilution. Both methods give results accurate to about 0.1%.

—J. H. L.

*Polysaccharides. VII. Constitution of cellobiose.* P. Karrer and F. Widmer. Helv. Chim. Acta, 1921, 4, 295—297.

*Hydrolysis of heptamethyl- $\beta$ -methylcelloside (J.,*

1921, 342A) by 5% hydrochloric acid yields tetra- and tri-methylglucoses, and since heptamethyl- $\beta$ -methylcelloside is obtained by methylation of cellobiose, the latter must, in accordance with the formulae for tri- and tetra-methylglucoses, be 5- $\beta$ -d-glucoside-d-glucose. This constitution has been given also by Haworth and Hirst (J., 1921, 234A), but the heptamethyl- $\beta$ -methylcelloside used by these authors contained an admixture of heptamethyl- $\alpha$ -methylcelloside.—T. H. P.

*Starch; Gelatinisation of — in cold water in presence of alkalis or neutral salts.* A. Reyher. Bull. Soc. Chim. Belgique, 1920, 29, 118—122.

STARCH grains may be rapidly gelatinised at the ordinary temperature by solutions of potassium or sodium hydroxide, but not by ammonia; by using a 0.75% solution of potassium hydroxide the process may be made sufficiently slow for microscopical observation. Many neutral salts also produce gelatinisation. Among the least active are sodium and calcium chlorides, whilst ammonium chloride, stannous chloride, mercuric chloride, lead nitrate, and sodium benzoate are more active, but even in concentrated solutions they act more slowly than 0.75% potassium hydroxide solution. The following substances at the concentrations stated have the same gelatinising power as 0.75% potassium hydroxide solution:—Sodium hydroxide at 0.53%, potassium iodide at 26—28%, ammonium nitrate at 30—35%, silver nitrate at 29%, potassium thiocyanate at 12—15%, and chloral hydrate at 55%. The mechanism of the gelatinisation process is discussed from the botanical standpoint.

—J. H. L.

*Amylopectin; The fundamental organic substance of —.* Samec and A. Mayer. Comptes rend., 1921, 172, 1079—1082.

A NEW classification is suggested for the products of hydrolysis of starch, namely, amyloses (without reducing power), dextrans (with reducing power), dextrinic acids (acid reaction), prefixing these names by amylo-, erythro-, or achroo-, according as the particular member gives a blue, red, or no colour with iodine. The carbohydrate derived from amylopectin would thus be termed an erythramylose.—W. G.

*Inulin; Application of the biochemical method of detection of dextrose to the study of the products of enzymic hydrolysis of —.* E. Bourquelot and M. Bridel. Comptes rend., 1921, 172, 946—949.

Using the method previously described (J., 1920, 344A), it is shown that the products of hydrolysis of inulin by the inulase of *Aspergillus niger* contain reducing sugars but no dextrose. Inulin is thus constituted by the condensation of laevulose molecules alone.—W. G.

#### PATENTS.

*Centrifugal apparatus for straining liquids [sugar juices].* J. Y. Johnson. From F. W. Fischlein. E.P. 161,822, 23.3.20.

THE apparatus, which is designed particularly for the straining of defecated sugar juices, consists of a casing within which is mounted on ball bearings a vertical shaft carrying one or more conical strainers. The shaft is rotated by means of a turbine at its upper end, to which the juice to be strained is delivered under pressure. After effecting the rotation of the turbine the juice falls from the latter into the top strainer, which preferably has the widest perforations, and passes in turn through the lower and finer ones. In each strainer is a deflector to direct the falling liquid against the conical walls, but a narrow annular opening near the bottom of each strainer permits solid matters retained by the latter to fall into the next strainer



and eventually to escape from the lowest strainer into a special protected outlet at the bottom of the casing. The casing has a separate outlet for the liquid.—J. H. L.

*Lactose or milk sugar; Manufacture of* — J. Tavroges, J. W. Roche, and G. Martin. E.P. 161,887, 22.7.20.

THE lactalbumin contained in whey from cheese manufacture is completely precipitated by means of a colloidal substance added to or produced in the whey. For example, the whey, heated to 70°–80° C., is treated with about 0.25% of sodium thiosulphate, which by reacting with the free acid of the whey produces a precipitate of colloidal sulphur; the latter, being negatively charged, precipitates the positively charged albumin. After removal of the precipitate the whey is concentrated for the recovery of lactose.—J. H. L.

*Caramel; Substitute for* — H. Thoms. G.P. 333,115, 1.4.20.

VEGETABLES which contain carbohydrates in any form, such as cellulose, lignocellulose, galactans, etc., are subjected to acid hydrolysis, e.g., by heating with dilute hydrochloric acid under atmospheric or higher pressures, and the product is evaporated to dryness and heated to 200° C. to caramelize the sugars. Remaining traces of acid and other odorous substances may be removed by a current of air. The residual brown mass is extracted with water and the filtered extract is concentrated.—J. H. L.

*Starch; Production of* — from potatoes. H. Wertheim; and Nyitraer Stärkeindustrie-A.-G. G.P. 331,378, 1.3.17. Conv., 3.2.17.

THE pulped potatoes are freed from juice as far as possible by known methods and then treated with the minimum amount of water necessary to remove the remaining extractive matters, and finally the starch is washed out with water as usual. The process renders possible the complete recovery of all valuable constituents of the potatoes, and avoids the difficulties associated with the treatment of waste waters obtained by the usual methods.—J. H. L.

*Sugar-cane mills*. C. McNeil. E.P. 161,726, 26.1.20.

See also pages (A) 405, *Sugar from wood* (E.P. 142,480); *Saccharification of cellulose* (E.P. 146,860); *Glucose from wood* (E.P. 143,212). 407, *Foods* (E.P. 161,885); *Food product* (G.P. 329,321).

## XVIII.—FERMENTATION INDUSTRIES.

*Fermentation; Researches on* — VII. Influence of additions (toluene, chloroform, thymol, and neutral salts) upon the fermentative decomposition of dipeptides by yeast extract. E. Abderhalden and A. Fodor. Fermentforsch., 1920—1, 4, 191–208.

EXPERIMENTS were carried out on the decomposition of glycyl-L-leucine by yeast extract, the course of the reaction being followed at intervals of 10 mins. by the formaldehyde titration method. Additions of toluene, chloroform, and thymol had no effect upon the decomposition, which is explained by the fact that these substances have no influence upon the colloidal state of the yeast enzymes. The addition of small amounts of sodium chloride caused a slight retardation in the reaction, which was increased by increasing the amount of salt added; whether the yeast extract were fresh or old made little material difference. With additions of potassium chloride, bromide, nitrate, and sulphate there was a slight acceleration of the reaction dur-

ing the first interval of time, then a slight retardation, and then a rapid inhibition. This result was given by a concentration of two-thirds an equivalent of each salt per l.; with higher concentrations the inhibition began earlier. Potassium iodide and thiocyanate produced at once a marked diminution in the reaction. It is concluded that the influence of the salt added is of a complex nature and depends upon the change produced in the electric charge of the solution and upon the change in the colloidal state produced thereby. The addition of the salts has the effect of displacing the hydrogen ion concentration of the solution towards the acid side of the scale.—J. H. J.

*Fermentation. VIII. Production of ferment sols from yeast phosphoprotein. Activity of the sols as a function of the colloidal state*. A. Fodor. Fermentforsch., 1920—1, 4, 209–229.

YEAST maceration extract prepared in the usual manner was treated with dilute hydrochloric acid when the phosphoproteins were precipitated; these were then washed with water and triturated with water to form sols, which in the absence of traces of acid were stable and fermented solutions of polypeptides. The sols obtained were examined ultramicroscopically and various degrees of dispersion were noticed. It was found that the fermentative activity varied directly with the degree of dispersion of the sol. The addition of alkalis and of alkali phosphates increased the degree of dispersion and the fermentative activity of the sols. The addition of potassium chloride in small amounts increased the fermentative activity, but in large amounts caused flocculation. Yeast phosphoproteins which have become more or less inactive can be regenerated, preferably by the addition of alkali and potassium chloride, with a corresponding increase in degree of dispersion.—J. H. J.

*Diasase; The co-enzyme (complement) of* — W. Biedermann. Fermentforsch., 1920—1, 4, 258–300.

ANIMAL and plant diastases consist of a thermostable and by itself inactive component (zymogen) and a thermostable co-enzyme, such as an inorganic salt. Many experiments were carried out to determine the effect of salts on diastatic fermentation and also on the influence of the reaction of the solution. It was found difficult to obtain by dialysis a completely inactive diastase solution, as traces of salts were sufficient to activate the enzyme. The salts used as activators were the neutral salts of the alkali and alkaline-earth metals with inorganic and organic acids, phosphates, bicarbonates, and thiocyanates. The anion proved to be the active component. The most active salt was sodium chloride, closely followed by potassium thiocyanate; in the second rank were potassium chloride and bromide, sodium bromide, and ammonium chloride; in the third rank were the chlorides of calcium, magnesium, strontium, and barium. After these came nitrates, iodides, and sulphates. Of the cations sodium had the most influence, followed by potassium. All the salts form with diastase complex compounds of indefinite composition which are easily dissociated. The activity of these neutral salt-diastases is greater in strictly neutral solutions; the slightest addition of acid and alkali is enough to check fermentation at once, and with long contact the enzyme is destroyed. Alkaline and acid salts behave differently from neutral salts. Acid sodium phosphate and acid sodium citrate in solutions up to 0.3% strength activate salt-free diastase in spite of the acid reaction of the mixture; above this strength the reaction is checked. Disodium phosphate and sodium bicarbonate activate fermentation in concentrations in which the more acid salts would inhibit. There is a relative retardation produced by disodium phosphate which



can be removed at once by a trace of sodium chloride, the reaction remaining neutral. The diastatic power of this mixture exceeds that of all others. The high activating power of phosphate ions is shown by the fact that the activating power of a sodium chloride solution is not only unimpaired but is considerably increased by a slight addition of a diphosphate. Similarly sodium carbonate does not cause any checking of an active sodium chloride-diastase. Saliva yields a diastase solution which depends for its activity upon the composition of the co-enzyme, that is, the combination of inorganic salts, the most important of which are sodium chloride, potassium thiocyanate and phosphate, to which must be added bicarbonate and carbon dioxide, which play an important part in the diastatic fermentation of pancreatic juice.

—J. H. J.

*Carboligase: an enzyme linking carbon chains.* C. Neuberg and J. Hirsch. *Biochem. Zeits.*, 1921, 115, 282—310.

By the fermentation of sucrose or dextrose by top yeast, bottom yeast, or maceration juice in the presence of benzaldehyde a hydroxy-ketone of the constitution,  $C_6H_5.CO.CH(OH).CH_2$ , or  $C_6H_5.CH(OH).CO.CH_2$ , is formed. Its production is attributed to the presence in yeast of a new enzyme, to which the name carboligase is given, which produces a condensation of the benzoin type between one molecule of benzaldehyde and one either of acetaldehyde or of pyruvic acid, its precursor in alcoholic fermentation. The same compound is formed by the action of yeast upon a mixture of benzaldehyde and pyruvic acid, but the direct condensation of the two aldehydes cannot be effected. (*Cf. J.C.S.*, June.)—E. S.

*Amygdalinase and amygdalase; Curious modification of — due to ageing.* G. Bertrand and A. Compton. *Bull. Soc. Chim.*, 1921, 29, 229—237.

Under the influence of time amygdalinase and amygdalase, as extracted from almonds, lose their activity very slowly, to the extent of about one-third in ten years, but they show a marked variation in the optimum hydrogen-ion concentration for the medium in which they act. Thus a sample of emulsin (amygdalinase+amygdalase) prepared in 1910 showed maximum activity in a medium slightly alkaline to phenolphthalein, but when examined in 1914 it showed maximum activity in a medium slightly acid to this indicator.—W. G.

*Enzymes of B. coli communis. V.—(a) Anaerobic growth followed by anaerobic and aerobic fermentation. (b) The effects of aeration during the fermentation.* E. C. Grey and E. G. Young. *Proc. Roy. Soc.*, 1921, 92 B, 135—150.

ANAEROBIC fermentation of dextrose by an emulsion of *B. coli communis* proceeds differently according as the organisms have been grown previously with or without oxygen. When the immediate past history has been anaerobic the fermentation under anaerobic conditions yields little or no lactic acid and a greatly diminished quantity of succinic acid. In place of these acetic acid appears in large proportion. Admission of oxygen during the fermentation leads to lactic acid production, there being an increase in lactic, acetic, and succinic acids and a diminution in hydrogen, carbon dioxide, and formic acid, the alcohol remaining practically constant. Under anaerobic conditions greater variations occur in the proportion of alcohol to acetic acid than under aerobic conditions, one of the effects of the introduction of oxygen during the fermentation being apparently to inhibit the mechanism of auto-reduction which is responsible for the variations in alcohol. Another

effect of the introduction of oxygen is probably to diminish the part played by water in the reactions.  
—W. G.

*Bacteria; Influence of fats on the growth of —.* H. Much and H. Schmidt. *Z. Immunitätsforsch., Exp. Ther.*, 1920, 31, 169—200. *Chem. Zentr.*, 1921, 92, I, 645.

THE addition of fats to nutrient media does not accelerate the growth of bacteria; with many species the growth is hindered, and chaulmoogra oil has a retarding effect on all bacterial growth. Staphylococci decompose the fats, and in a fatty medium the organisms themselves appear to be somewhat larger than usual and to a certain extent swollen, but their other properties remain unaltered, except when chaulmoogra oil is present.

—W. P. S.

*Fruit [for wine making]; Influence of degree of ripeness of — on the acceleration of fermentation by various nitrogenous compounds. Cellar experiments to secure pure fermentation and stability of fruit wines. Lactic acid malady appearing in fruit wines after complete fermentation of the sugar. Importance of blending for the stability of mild fruit wines.* H. Müller-Thurgau and A. Osterwalder. *Landw. Jahrb. der Schweiz*, 1920. *Chem. Zentr.*, 1921, 92, II, 748—750.

EXPRESSED juices from unripe, ripe, and over-ripe pears of four kinds were fermented with and without addition of ammonium chloride or carbonate, and with and without the use of pure culture yeast. The effect of ammonium salts confirmed previous conclusions (J., 1919, 333 A); they accelerated the fermentation of juice from unripe pears less than that of juice from ripe pears, and it is concluded that the former already contain more assimilable nitrogen than the latter. Spontaneous fermentation was in all cases sluggish, but with addition of pure yeast the juices from unripe fruit fermented at least as rapidly as those from ripe fruit, the higher tannin-content of the former having no perceptible retarding influence. Most fruit wines, especially those of low acidity and tannin-content, require more careful treatment in the cellar than grape wines. A common malady is one in which lactic and acetic acids are formed, and unlike ordinary acetous fermentation this cannot be checked by exclusion of air. The best preventive measures are rapid fermentation, i.e., use of pure yeast and ammonium salts, and avoidance of high temperatures. Treatment with sulphur dioxide or metabisulphite is desirable to prevent the development of the malady after fermentation. The malady is distinct from that in which mannitol and lactic and acetic acids are formed from sugar by *Bact. mannitolipacum*; in the former the acids appear to be produced from glycerol and other unknown constituents, for it may develop after all the sugar has been fermented. In the case of juices of low acidity and tannin-content the malady may be warded off during fermentation by mixing such juices, before fermentation, with apple or pear juices rich in acid and tannin (pear juices rich in tannin alone are not so suitable). The mixed juice is fermented as rapidly as possible and afterwards sulphited.—J. H. L.

*Maize silage.* Fred and others. See XIXa.

#### PATENTS.

*Acetone and higher alcohols, particularly butyl alcohol; Method and apparatus for the production by fermentation of —.* A. Fernbach. E.P. 109,969, 30.8.17. Conv., 31.8.16.

THE method described in E.P. 15,203, 15,204, and 16,925 of 1911, and 21,073 of 1912 (J., 1913, 987),

may be carried out in apparatus of the kind described in E.P. 15,561 of 1898 (J., 1898, 1066); this apparatus comprises fermentation vats each provided with a feeding orifice, an agitator, a system of pipes for the introduction of air or inert gas, communicating pipes, and a filter. A small "leavening vat" is used in conjunction with the fermentation vats, and means are provided by which the reduced pressure resulting from the cooling of the sweet wort after sterilisation is utilised for introducing the culture.—W. P. S.

*Wood and other cellulose-containing substances; Manufacture of sugar and furfural from —.* A. Classen. E.P. 142,480, 27.4.20. Conv., 18.3.19.

THE material is heated under a steam pressure of about 7–8 atm. with sufficient dilute sulphuric or hydrochloric acid to effect conversion, but one or more additional acids, or salts capable of yielding acids, are employed at the same time as catalysts. The action of the additional acids improves the yield and fermentable quality of the sugar produced, and the yield of furfural can be increased by a suitable choice of additional acids, e.g., sulphuric and sulphurous acids when hydrochloric acid is used for conversion, or hydrochloric and sulphurous acids when sulphuric acid is used for conversion. Besides acid catalysts metals or metallic oxides, especially those of the iron group, may be employed.—J. H. L.

*Glucose [for fermentation] from wood; Process and apparatus for obtaining —.* H. Terrisse and M. Levy. E.P. 143,212, 6.5.20. Conv., 13.5.19.

Dry sawdust is treated in a mixing cylinder with about twice its weight of 33% hydrochloric acid and is then conveyed through other mixing cylinders into each of which gaseous hydrochloric acid is introduced so as to increase the acid concentration to about 41%. During these operations the temperature is maintained at 0°–25° C. The impregnated material is digested in a rotating vessel at a slightly higher temperature for 10 hrs., and the excess of hydrochloric acid is removed by raising the temperature to about 30° C. and reducing the pressure to 40 mm., the gaseous acid being absorbed and collected. The residual mass is extracted with water, the extract filtered and distilled under reduced pressure to remove hydrochloric acid completely.—W. P. S.

*Cellulose-containing materials; Saccharification of —.* A. Wohl. E.P. 146,860, 5.7.20. Conv., 7.4.17.

THE material is intimately mixed with strong mineral acid, e.g., 70–75% sulphuric or 39–40% hydrochloric acid, and subjected to powerful agitation and compression. The amount of acid required is much less than when the material is not kneaded and pressed, e.g., 1.5 pt. of 75% sulphuric acid is sufficient for 1 pt. of wood waste.—J. H. L.

*Cellulose; Fermentation of —.* The Power-Gas Corporation, Ltd., and H. Langwell. E.P. 161,294, 8.1 and 18.3.20. Addn. to 134,265 (J., 1920, 38 A).

IN the process described (*loc. cit.*) acetic acid is the chief product when the aeration is limited; if the rate of aeration is increased, alcohols, particularly ethyl alcohol, are the principal products of the fermentation.—W. P. S.

*Yeast; Manufacture of pressed — from distillers' grains, oil-cake and the like.* J. Effront. E.P. 160,496, 21.11.19.

NITROGENOUS material such as oil-cake, distillers' grains, etc., is digested with concentrated hydrochloric or other mineral acid for 48 hrs. at 106°–108° C. The extract obtained, which is rich in

amino-acids, is employed as yeast food, whereby the amount of sugar consumed by the yeast without production of alcohol is much reduced.—J. H. L.

*Fermented liquors; [Use of sour whey solids in the] manufacture of —.* C. S. Townsend. E.P. 160,562, 23.12.19.

THE solids obtained by the desiccation of sour whey from the manufacture of cheese are employed as a constituent of fermented beverages, e.g., "stone ginger beer." They assist fermentation and give a distinctive flavour to the beverage. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 20,183 of 1912; J., 1913, 987.)—J. H. L.

*Fermentation process.* A. Römer, and Deutsch-Koloniale Gerh- u. Farbstoff-Ges. m.h.H. E.P. 161,870, 17.6.20.

IN a continuous process for the fermentation of saccharine liquids, e.g., molasses worts or sulphite-cellulose liquors, the liquid is passed through a series of "ferment filters," i.e., cylindrical vessels containing yeast fixed in kieselguhr, sawdust, or the like. The liquid is passed upwards through each vessel, at a suitable temperature and at such a rate that after traversing the whole series, e.g., 6 vessels, it is completely fermented. The order of the vessels may be changed at will so that the yeast in any one does not remain too long in contact with liquid poor or rich in sugar. Any vessel may be cut out of the series, re-charged with fresh yeast, and brought into operation again without arresting the working of the others. For the fermentation of liquids poor in sugar one filter may suffice. The process may be adapted to other types of fermentation, e.g., to the production of butyl alcohol, glycerol, lactic, butyric, or citric acid, acetone, etc.—J. H. L.

*Mashing process.* H. E. Deckebach. U.S.P. 1,374,191, 12.4.21. Appl., 20.9.19.

GROUND malt is mashed in a mash tun and raw grits are simultaneously heated with water in a converter, and the two vessels are so connected that the materials can be circulated from the tun into the converter and from the converter into the tun, until the starch is saccharified.—J. H. L.

*Fermentation of beer and the like [by the Burton union system]; Apparatus for —.* J. F. Warwick. E.P. 161,496, 16.8.20.

*Caramel substitute.* G.P. 333,115. See XVII.

*Foods.* E.P. 161,885. See XIXA.

## XIXA.—FOODS.

*Milk; Relation between lactic acid production and bacterial growth in the souring of —.* J. C. Baker, J. D. Brew, and H. J. Conn. New York Agric. Expt. Stat. Bull. No. 74, 1919. 24 pages.

THE rate of acid production per individual cell per hr. with different cultures of bacteria, varied considerably, being much lower in the case of an organism which multiplied arithmetically than with one which multiplied normally (geometrically); for a vigorous culture the rate was between  $5 \times 10^{-10}$  and  $10 \times 10^{-10}$  mg. of lactic acid per hr. The ratio of total acid produced to the number of organisms present proved to be fairly constant, and was directly proportional to the amount of acid produced per generation by each individual cell; the ratio increased as the curdling point was approached. There was a tendency for the acid present to increase geometrically until coagulation ensued and, to a certain extent, this was true even with the organisms which were multiplying arith-

metically. At the curdling point, the multiplication of the bacteria appeared to cease and there was a noticeable decrease in the rate of acid production. The bacteria were, in all cases, counted individually under the microscope.—W. P. S.

*Milk powders; Antiscorbutic potency of* —. E. B. Hart, H. Steenhock, and N. R. Ellis. *J. Biol. Chem.*, 1921, 46, 309—318.

APART from the original content of vitamin, as influenced by fodder, the powders vary with the process of manufacture used; the spray processes are more destructive than the Just (heated rolls) process. With the possible exception of powders made by the Just process from summer milk (or even from winter milk obtained on a vitamin-rich diet of roots and tubers) these preparations must be supplemented in infant feeding by some potent source of antiscorbutic vitamin.—G. B.

*Vitamin; Stability of the antiscorbutic* —, and its behaviour to various treatments. N. R. Ellis, H. Steenbock, and E. B. Hart. *J. Biol. Chem.*, 1921, 46, 367—380.

THE antiscorbutic vitamin in cabbage is destroyed by desiccation at 65° C., even in an atmosphere of carbon dioxide, and also by the fermentation involved in the making of sauerkraut, and of silage from maize. It is not removed from orange juice by ether or by aeration, but is destroyed by oxidising agents. Blood charcoal and the Chamberland filter remove a measurable amount from orange juice.—G. B.

*Fats; Special nutrition values of different* —. Accessory food factors. I. H. Aron and R. Gralka. *Biochem. Zeits.*, 1921, 115, 188—203.

FEEDING experiments on rats are described which confirm the presence of vitamin A in cod-liver-oil, butter, and egg-yolk, and its absence in margarine.—E. S.

*Alimentary ration and vitamins.* A. Desgrez and H. Bierry. *Comptes rend.*, 1921, 172, 1068—1071.

ON a ration, deprived of fats, containing only protein and carbohydrates, but rich in vitamins, the growth of young rats and the equilibrium of adult animals can be assured, but in the complete absence of vitamins, nitrogen equilibrium can only be maintained if the constituents of the ration are all present and in certain proportions.—W. G.

*Egg preparations; Drying and preserving of* —. Briant and Harman. *Nat. Assoc. Review*, 1921, 38, 247—251.

THE haking value of eggs lies in their power of retaining gas produced by haking powders etc. in the dough, and the value is dependent on the amount of coagulable albumin present. In the case of dried eggs a portion of the albumin may be coagulated during the drying process, especially if the temperature exceeds 65° F. (19° C.), and the loss due to coagulation is increased by the presence of small quantities of boric acid.—W. P. S.

*Lactic acid bacteria; Relation of* — to corn [maize] silage. E. B. Fred, W. H. Peterson, and J. A. Anderson. *J. Biol. Chem.*, 1921, 46, 319—327.

*Lactobacillus pentoaceticus* plays an important part in maize silage and increases the amount of alcohol and volatile acid, while the lactic acid is decreased. *Bacillus lactis acidii* persists only during the first days of fermentation.—G. B.

*Pentosans; Destruction of* — in the formation of silage. W. H. Peterson, E. B. Fred, and J. H. Verhulst. *J. Biol. Chem.*, 1921, 46, 329—338.

CORN (maize) fodder contains 21.8% of pentosans

and 0.34% of methylpentosans. After fermentation for 50 days the content of pentosans ranges from 17.8 to 20.9% according to the moisture present. Taking account of the loss of dry matter, at least 15—20% of the pentosans is destroyed during fermentation. Pentoses or other furfural-yielding substances soluble in water occur, probably as the result of bacterial action. A mixture of 2% of acetic and 5% of lactic acid did not appreciably hydrolyse pentosans in 20 days at 28° C. Immature maize contains 0.6% of free pentosans.—G. B.

*Silage crops; Application of the Van Slyke method to hydrolysed protein extracts of* —. R. E. Neidig and R. S. Snyder. *J. Amer. Chem. Soc.*, 1921, 43, 951—959.

THE proteins of forage crops, either before or after conversion into silage, cannot be successfully analysed by the Van Slyke method applied directly to the hydrolysed extract of the original material, because of the formation of considerable quantities of humin from the cellulose. The method of Eckstein and Grindley (*J.*, 1919, 301A) gives results which permit of a fair interpretation of the protein molecule when applied to concentrates which contain but little cellulose.—W. G.

*Carnauba wax palm.* Grimme. See XII.

*Caffeine in yerba maté etc.* Ugarto. See XX.

*Alkaloids in cacao.* Ceriotti. See XX.

*Protein analysis.* Menaul. See XXIII.

#### PATENTS.

*Grape honeys and fruit syrups; Process and apparatus for the manufacture of* —. E. Barbet et Fils et Cie. E.P. 153,548, 21.1.20. Conv., 31.10.19. Addn. to 135,175 (*J.*, 1921, 364A).

THE apparatus previously described (*loc. cit.*) has the disadvantage that the vapours given off from the first evaporation are sometimes charged with sulphurous acid, which slowly attacks the copper and may thereby contaminate the juice. This is obviated by subjecting the sulphited juice to a preliminary desulphitation in a tower used in conjunction with each of the evaporating groups of the earlier apparatus and made of ceramic ware or vulvic lava. The desulphitation is effected by means of juice vapour introduced into the bottom of the tower through a regulating valve. The desulphited liquor passes from the bottom of the tower to the evaporator.—G. F. M.

*Eggs; Method of preserving* —. G. Dreyer and G. F. Hanson. E.P. 161,110, 21.7.20.

EGGS are immersed in, or washed with, formaldehyde solution or with water containing chlorine, and are then stored in a receptacle through which a current of moist sterilised air at 5° C. is passed. The eggs are turned once every 24 hrs. during storage.—W. P. S.

*Alimentary products containing water-soluble vitamins; Manufacture of* —. "Forget-me-not-Flours," Ltd., and R. Hutchinson. E.P. 161,238, 23.12.19.

WHEAT bran, or the husk of other edible seeds, is dried at 100° F. (38° C.) under reduced pressure and ground; the powder may be used as such or as aqueous infusion may be prepared from it. The infusion may be mixed with gelatin etc., and formed into tablets, or it may be evaporated under reduced pressure to produce a dry powder. Filtration of the infusion is optional.—W. P. S.

*Food preparation [from pine seeds]; Production of a* —. C. Farmachidis. E.P. 161,291, 8.1.20.

SHELLED pine seeds are washed, dried, ground, and

mixed with flour and gluten; other substances, such as baking powder, sugar, flavouring matter, etc., may be added. The mixture is formed into biscuits, which are baked at 130° C.—W. P. S.

*Vegetable gelatin; Process of making [edible] — and the product thereof.* M. M. Malcolm and C. S. Townsend. E.P. 161,612, 10.11.19.

VEGETABLE gelatin is prepared from seaweed, especially from *Chondrus crispus* or carrageen, by soaking the material in water for some hours, washing to remove impurities and salts, bleaching the softened substance with "floridin," "norit," or albumin, disintegrating by boiling with 5 pts. of water containing acids or alkalis, preferably 0.5% acetic acid, with gentle agitation, and finally expressing the liquid from the undissolved residue. A preservative such as 0.5% of bisulphite, or 1.0% of sodium benzoate, is added to the liquor, and the gelatin may be dried by running the filtered material on to heated rollers, to produce sheets; by atomising the liquor after a preliminary concentration, into a chamber through which a current of hot air is passed, whereby a gelatin powder is produced, or by drying the concentrated liquor in trays in an oven to produce the material in block form.—G. F. M.

*Foods; Process for the preservation of by-products of brewing, distillation and other processes for use as animal —.* A. Stouffs. E.P. 161,885, 19.7.20.

BY-PRODUCTS suitable for feeding-stuffs, e.g., brewers' grains, exhausted beet slices, etc., may be preserved from fermentation for about 10 months by moistening them with dilute hydrochloric acid at the rate of 1 kg. of 33% fuming acid diluted with 200 l. of water per 1000 kg. of material. The products may be compressed into cakes.—J. H. L.

*Meat; Process of producing powdered —.* J. C. MacLachlan, Assr. to Standard Food Products Co. U.S.P. 1,372,527, 22.3.21. Appl., 26.5.19.

FINELY-CUT or ground meat is projected by centrifugal force into a hot drying medium, in which it is intercepted, beaten, and desiccated.—J. H. L.

*Cheese; Process of sterilising —.* E. E. Eldredge. U.S.P. 1,374,141, 5.4.21. Appl., 24.9.19.

CHEESE is mixed with 2% of its weight of sodium phosphate, and then heated at a suitable temperature.—W. P. S.

*Sugar; Composition of — and method of preparing the same.* N. C. Fowler, jun.; F. A. Fowler, adminatrix. U.S.P. 1,374,160, 5.4.21. Appl., 8.8.16. Renewed 16.4.18.

DRIEN fruit is ground with the addition of water and sugar, the mixture dried, and then re-ground.—W. P. S.

*Milk; Production of dehydrated —.* S. M. Dick, Assr. to International Dry Milk Co. U.S.P. 1,374,555, 12.4.21. Appl., 31.1.20.

MILK is atomised without pressure in a suitable dehydrating cell, and is exposed to the action of compound currents of heated air. It is thereby dried without breaking or injuring the globules of butter fat, which become coated with casein, albumin, and other colloidal constituents of the milk. The product is readily soluble with the production of a liquid substantially the same as normal milk, on which the cream will rise in the usual way.—G. F. M.

*Food product; Manufacture of a — [from sugar residues or the like].* H. Stoltzenberg. G.P. 329,321, 24.4.19.

RESIDUES from the desaccharification process, e.g.,

osmose molasses, wash liquors, etc., are treated hot with an organic solvent, such as alcohol or a ketone or ester, and after the mixture has cooled and stood for some time, the upper layer, i.e., the organic solvent, which contains the unwholesome constituents in solution, is separated. After the lower layer has stood for a further period, the amino-acid salts which settle out are removed, converted to the free acids, and returned to the solution, which has in the meantime been freed from the last traces of organic solvent by heat. The product can be employed as a food product or as fodder, or can be fermented to yield an alcoholic beverage or for the production of pressed yeast, or it can be utilised in the manufacture of sweetstuffs and the like.

—L. A. C.

*Rennet, rennet extracts and the like; Process for the preparation of —.* Gebr. Schubert. G.P. 333,453, 5.6.18.

THE material is fractionally extracted with alcoholic calcium chloride solution and the united extracts evaporated. The use of calcium chloride allows of the concentration of weak extracts without precipitation of the salts or the enzyme.—A. de W.

*Food from fish-flesh; Preparation of —.* M. Jewnin. G.P. 333,554, 8.4.19.

THE comminuted flesh is extracted with aqueous alcohol of a strength not exceeding 70%, and is then mixed with amino-acids resulting from the acid hydrolysis of proteins and with flour. The product is free from the characteristic, disagreeable fishy flavour.—A. de W.

*Fodder; Process for the conversion of the stomach-contents of animals into a dry —.* G. Beckstroem. G.P. 334,170, 17.6.19. Addn. to 300,063 (J., 1919, 960 A).

FISH or fish waste may be employed together with stomach-contents and cellulosic matter. The bacteria which develop attack fish bones, scales, and fins.—J. H. L.

*Desiccating buttermilk, sour milk, and the like; Apparatus for —.* Collis Products Co., Assees. of N. P. Collis. E.P. 133,092, 20.1.20. Conv., 13.12.15.

SEE U.S.P. 1,317,777 of 1919; J., 1919, 960 A.

*Desiccating buttermilk and the like.* A. E. White. From Collis Products Co. E.P. 161,678, 13.1.20.

SEE U.S.P. 1,356,340 of 1920; J., 1920, 831 A.

*Cooler [for edible fat mixtures].* W. Clayton and G. Nodder. U.S.P. 1,375,210, 19.4.21. Appl., 26.3.20.

SEE E.P. 155,477 of 1919; J., 1921, 126 A.

*Specific gravity tester [for cooked food products].* J. H. Kessler. E.P. 162,240, 18.11.20.

*Treating colloid-containing mediums.* U.S.P. 1,359,037. See VII.

*Lactose.* E.P. 161,887. See XVII.

*Fermented liquors.* E.P. 160,562. See XVIII.

## XIXB.—WATER PURIFICATION; SANITATION.

*Water; Determination of the hydrogen ion concentration of —.* I. M. Kolthoff. Z. Unters. Nahr. Genussm., 1921, 41, 112—122.

A COLORIMETRIC method for the determination of the hydrogen ion concentration of water consists

in treating the water with Neutral Red solution and comparing the coloration with that of standardised acid and alkaline solutions of the same indicator contained in graduated wedge-shaped tubes. The carbon dioxide content of a water may be calculated from the hydrogen ion concentration and the bicarbonate content by the formula  $[H_2CO_3] = [H^+] [HCO_3^-] / 3.04 \times 10^{-4}$ .—W. P. S.

**Water; Influence of minute quantities of metallic salts in — on its bacteriological content.** E. L. Atkinson and R. O. Frederick. J. Roy. Nav. Med. Service, April, 1921. [Reprint.] Pp. 5.

NATURAL waters of different types were treated with lead acetate, copper sulphate, ferric chloride, or zinc sulphate, equivalent to 0.5 and 0.25 pt. of metal per 100,000, and after inoculation with *B. typhosus* each sample was sub-cultured into ordinary nutrient broth tubes on four successive days. The results show that even 0.25 pt. of copper per 100,000 in the water markedly inhibited development in the cultures; zinc had a slight effect at 0.5 pt. per 100,000 but practically none at 0.25 pt., whilst lead and iron at 0.5 pt. per 100,000 were without any appreciable action. In further experiments an upland surface water and a mixture of shallow well and upland surface waters were treated with salts as above, and after standing for 48 hrs. were subjected to routine bacteriological analysis. Here again the results were seriously affected even by 0.1 pt. of copper per 100,000 of water, and lead and zinc at 0.5 pt. per 100,000 appeared to influence the results in some cases. The authors conclude that a chemical analysis of water is necessary for the interpretation of the results of bacteriological tests.—J. H. L.

**Nitrogen; Conservation of — with special reference to activated [sewage] sludge.** G. J. Fowler. J. Indian Inst. Sci., 1920, 3, 227—279.

AN extensive review is given of the existing data as to the nature and amount of the nitrogen in faeces and sewage and of the attempts which have been made to recover some of its value for agricultural purposes. In the activated sludge process of sewage purification there appears to be an increase of nitrogen in the end products over the quantity in the crude sewage treated. Laboratory experiments in which activated sludge and sewage were agitated together by means of an air diffuser, the clarified liquid being withdrawn daily for 5 days and fresh sewage added, showed that the sum of the total nitrogen in the original sludge and in the sewage added daily amounted to 1.793 g., and that in the effluent withdrawn daily and in the final sludge amounted to 1.865 g., an increase of about 4%. In experiments on the incubation of activated sludge with culture solutions favourable for the growth of nitrogen-fixing bacteria, there was found to be a gain of nitrogen of 25% in 4 weeks. For the purpose of studying the effect of activated sludge and the products of its decomposition on plant growth, a seedling was grown in sand partially immersed in activated sludge and water. Aeration was continued for 3 weeks when the liquid contained, per 100,000 pts., 0.03 pt. of ammonia and 0.2 pt. of nitrite, whereas a control experiment with sludge and air showed only 0.5 pt. of ammonia and 0.05 pt. of nitrite, and another with plant and sludge only 2.8 pts. of ammonia and no nitrite. The experimental plant grew well, whereas the control plant gradually died. It is suggested that the stimulating effect of activated sludge on plant growth may be due to the presence of auximones.

—J. H. J.

See also pages (A) 375, *Corrosion of boiler tubes* (Worth); *Rust formation* (Sauer). 379, *Charcoal*

*for gas masks* (Hawley). 388, *Adsorption by carbon* (Herbst). 401, *Soil fumigation* (Peterson). 413, *Trinitrotoluene poisoning* (Voegtlin and others). 414, *Toxicity of parazol* (Voegtlin and others); *Mercury fulminate* (Livingston).

#### PATENTS.

**Carbon dioxide; Removal of — from liquids, e.g., water for industrial purposes.** J. Muchka. G.P. 333,569, 28.2.19. Conv., 18.2.19.

WATER is heated above 100° C. and then passed in a fine spray into a small de-gassing chamber completely separate from the heating chamber. This process involves less loss of heat and steam than that of heating under pressure followed by agitation.—C. I.

**Fungicide and insecticide and method of producing the same.** D. S. Pratt. U.S.P. 1,374,951, 19.4.21. Appl., 11.10.19.

A LIME-SULPHUR solution is sprayed as a fine mist into a heated chamber and the vapour formed is withdrawn from the chamber. The solid is deposited as a fine, uniform powder containing a high proportion of the active ingredient readily soluble in cold water.—L. A. C.

**Insect lime.** Badische Anilin u. Soda Fabrik. G.P. 334,576, 12.9.19.

By the condensation of sulphonic acid chlorides with mixtures of isomeric aromatic amines, permanently sticky products are obtained which are unchanged by moisture, air, or weather conditions, and which either alone, or in combination with oils or solid materials such as chalk etc., can be utilised as insect lime.—G. F. M.

**Air; Method of producing exchange materials for purification of —.** C. Clemente. E.P. 138,649, 6.2.20. Conv., 22.6.16.

SEE G.P. 305,066 of 1916; J., 1919, 876 A.

**Sulphur; Manufacture of oxygen compounds of — [for use as disinfectants].** T. A. Clayton. E.P. 161,439, 9.4.20. Addn. to 141,661.

**Effluent or other liquids; Apparatus for treating — and for the separation and recovery of matters of different densities.** L. Linden. E.P. 161,735, 23.1.20.

#### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Caffeine in yerba maté, coffee, tea, kola nut, and guaraná; Determination of —.** T. Ugarte. Anal. Soc. Quim. Argentina, 1920, 8, 413—415.

HALF a gram of the finely powdered substance is introduced into a 500 c.c. flask, and carbonised by heating with a small flame. Dense white vapours are evolved and the heating is so regulated that they do not reach to the upper part of the neck of the flask. After cooling, the contents of the flask are washed out with successive 5 c.c. portions of water and filtered. The united filtrate is evaporated to dryness on a water bath and the residue redissolved in 2 c.c. of water. A few drops of N/1 ammonia solution and 5 c.c. of chloroform are added. After agitating, the two liquids are filtered through filter paper impregnated with chloroform and washed with two 5 c.c. portions of chloroform. The filtrate and washings are evaporated to dryness. Caffeine is extracted from the residue with successive small quantities of water and weighed after drying at 100°—105° C.

—G. W. R.

Cacao; Determination of alkaloids in —. A. Ceriotti. Anal. Soc. Quim. Argentina, 1920, 8, 400—403.

TEN grams of the fat-free material is boiled for at least 1 hr. with 150 c.c. of 5% sulphuric acid under a reflux condenser. Colouring matters, tannins, and resins are then precipitated by addition of 200 c.c. of 3% mercuric acetate, and the boiling is continued for another 10 mins. The filtrate after neutralisation with magnesia is evaporated to 50 c.c. on a water bath. After addition of 10 g. of sand and excess of calcined magnesia the liquid is evaporated to dryness. The alkaloids are extracted from the powdered residue in a Soxhlet apparatus by a mixture of benzene and chloroform (1:1) and, after removal of the solvents, weighed in a tared dish. The weight of theochromine is obtained by difference after three successive extractions with carbon tetrachloride to remove the caffeine.

—G. W. R.

Alkaloids of valerian. A. Goris and C. Vischniac. Comptes rend., 1921, 172, 1059—1061.

WALISZEWSKI's statement (Union Pharm., 1893, 34, 251) as to the existence of the two alkaloids chatinine and valerine in valerian root is confirmed. From 1 kg. of fresh root 0.1 g. of mixed alkaloids was obtained, the chatinine predominating. Chatinine is soluble in ether, whereas valerine is insoluble in ether but soluble in chloroform.—W. G.

Alkaloids; Behaviour of phospho- and silico-tungstic acids towards —. A. Heiduschka and L. Wolf. Schweiz. Apoth.-Zeit., 1920, 58, 213—218, 229—233. Chem. Zentr., 1921, 92, II., 840—841.

THE precipitates obtained by the addition of silico-tungstic acid to alkaloidal solutions are colloidal with the exception of the quinine and caffeine precipitates. The addition of electrolytes causes coagulation. An equilibrium exists between the neutral and acid salts, depending on the concentration of the acid during precipitation and during washing. By using N/100 solutions of silicotungstic acid in presence of 1% hydrochloric acid, sparteine, quinine, cinchonine, and nicotine are precipitated as di-acid bases corresponding to the formula  $\text{SiO}_2 \cdot 12\text{WO}_3 \cdot 2\text{H}_2\text{O} \cdot 2\text{Alkaloid} \cdot x\text{H}_2\text{O}$ , whilst the composition of the precipitates obtained with morphine, codeine, thebaine, and cocaine is represented by  $\text{SiO}_2 \cdot 12\text{WO}_3 \cdot 2\text{H}_2\text{O} \cdot 4\text{Alkaloid} \cdot x\text{H}_2\text{O}$ . In other cases the precipitate has no simple composition, and a quantitative determination is accordingly impossible in consequence of the alteration in composition by washing. Alkaloids forming silicotungstates which are only slightly altered by 0.1% sodium chloride solution can, in the absence of albumin, be determined by adding an excess of silicotungstic acid standardised with sodium hydroxide and methyl red, followed by 1% sodium chloride, filtering and washing the precipitate with 0.1% sodium chloride solution, and titrating back the excess acid in the filtrate and washings. Phosphotungstic acid behaves in a similar manner towards alkaloids. In presence of less than 1% of hydrochloric acid, sparteine, quinine, cinchonine, and nicotine give precipitates corresponding to the formula  $2\text{P}(\text{W}_2\text{O}_7) \cdot \text{H}_2\text{O} \cdot 3\text{Alkaloid} \cdot x\text{H}_2\text{O}$ , in other cases the composition is dependent on external conditions. The solubility of the alkaloidal silico- and phosphotungstates is affected to varying extents by the addition of hydrochloric acid or sodium chloride, but minute quantities of alkaloids can be determined with some degree of exactitude by comparing the opalescence produced by silicotungstic acid with that produced in a solution of known strength.

—G. F. M.

Hydrocyanic acid; Micro-crystalline reaction for the identification of — in cyanogenetic glucosides. G. Denigès. Comptes rend. Soc. Biol., 1921, 84, 309—310. Chem. Zentr., 1921, 92, II., 623—624.

THE substance to be tested is ground with water and, after some time, a portion is placed in a short, narrow test-tube; the mouth of the latter is covered with a microscope slide carrying on its under side a drop of alloxan reagent which has been rendered ammoniacal previously. The alloxan reagent is prepared by dissolving 1 g. of uric acid in a mixture of 1 c.c. of nitric acid (sp. gr. 1.40) and 1 c.c. of water and diluting to 50 c.c. If hydrocyanic acid is present, star-shaped crystals of oxaluramide are formed in the drop of reagent, usually within a few minutes. The test is more sensitive when pyridine is used in place of ammonia; in this case the crystals are in the form of bundles. It frequently happens that the reagent mixture shows a red coloration due to the simultaneous formation of murexide.—W. P. S.

Quinic acid; Presence of — in the leaves of some conifers. G. Tanret. Bull. Soc. Chim., 1921, 29, 223—229.

THE author has isolated quinic acid from the leaves of cedar trees growing in the neighbourhood of Paris, finding 5 g. of the crystallised acid per kg. of leaves (dried at 100° C.) picked in July, but only 2 g. per kg. in leaves picked in December. Similar results were obtained with larch leaves, but the acid could not be isolated from leaves of yew, juniper, or fir trees. The addition of ammonium molybdate causes marked modifications in the rotatory power of quinic acid. With increasing quantities of ammonium molybdate the value of  $[\alpha]_D^{20}$  (–43.2°) first increases to a maximum (–63°), then falls to a minimum (–54.8°), and finally increases again to a maximum limit value (–71.7°).—W. G.

Capsella bursa pastoris; Active constituents of —. H. W. van Urk. Pharm. Weekblad, 1921, 58, 553—556. (Cf. Boruitau and Cappenberg, J., 1921, 276 A.)

ALKALOIDS and allyl mustard oil are absent, and the presence of glucosides is very doubtful. (Cf. J.C.S., June.)—S. I. L.

Bromural. E. Yoshitomi and K. Watanabe. Yakugakuzasshi (J. Pharm. Soc. Japan), 1921, [463], 125—130.

PURE  $\alpha$ -monobromoisovalerylurea (bromural) melts at 154° C., whilst commercial samples melted at 145°—157° C. The difference is due mainly to the presence of methylethylbromouacetylurea and iso-valerylurea. The melting-point curve of mixtures of bromural and iso-valerylurea has a minimum, and hence the same melting point may correspond to two different mixtures. To ascertain the purity of bromural, determination of the melting point is, therefore, of no value, and the bromine content should be determined.—K. K.

Hydrogenation; A catalytic method of —. J. Nivière. Bull. Soc. Chim., 1921, 29, 217—219.

THE material to be hydrogenated, dissolved in a suitable solvent, is placed in a stout-walled bottle. A small amount of a solution of palladium chloride or platinum chloride is added to act as a catalyst, and then the calculated amount of calcium hydride is added gradually, the flask being stoppered and well shaken. This method was used successfully for the reduction of benzaldehyde, nitrobenzene, and benzylidene-acetone.—W. G.

Phenylhydrazones; Catalytic hydrogenation of —. A. Mailhe. Comptes rend., 1921, 172, 1107—1110.

PHENYLHYDRAZONES are hydrogenated when passed



in a rapid current of hydrogen over reduced nickel at 180°–190° C. If derived from aldehydes they give aniline and the corresponding nitrilo, but if derived from ketones they yield aniline, the primary amine, and some secondary amine.—W. G.

**Benzoic acid; Synthesis of chlorine-free — from benzene.** R. H. McKee and F. A. Strauss. Chem. and Met. Eng., 1921, 24, 638–644, 697–702.

THE reactions involved in the conversion of sodium benzenesulphonate into benzoic acid were investigated with a view to their utilisation on a commercial scale. Experiments on the direct formate fusion method indicated that benzoate formation only takes place at a high temperature and is accompanied by much decomposition. Synthesis through benzonitrile gave greater promise of success on the technical scale. The main reaction in which benzonitrile is formed is accompanied by side reactions of two general types—reduction of the sodium benzenesulphonate by sodium cyanide, and decomposition of the cyanate formed, by water vapour, with formation of sodium carbonate, carbon dioxide and ammonia, and secondly pyrogenic decomposition of the benzenesulphonate with production of a large number of substances including thioethers, thiophenols, hydrogen sulphide, etc. The most favourable reaction conditions for the formation of the maximum yield of benzonitrile were shown to be: Temperature of the reaction mixture must be kept between the limits of 420°–430° C.; finely powdered cyanide and sulphonate intimately mixed in equimolecular proportions, or a slight excess of cyanide; addition to the mixture of sand or other inert material, an admixture of 70% of 40-mesh sand being the optimum. Under these conditions a nitrile formation efficiency of about 43% was obtained. Reducing the pressure did not improve this result, but rather favoured the formation of sulphur compounds and tar. Hydrolysis of benzonitrile by sulphuric acid (sp. gr. 1.6) was more rapid and efficient than alkali hydrolysis, and gave yields amounting to about 95% of the theory of practically pure benzoic acid. With a source of cheap cyanide the authors consider that the process proposed would produce benzoic acid of a higher degree of purity and at a lower price than the methods at present in use.—G. F. M.

**p-Aminophenol; Electrolytic manufacture of —.** A. S. McDaniel, L. Schneider, and A. Ballard. Trans. Amer. Electrochem. Soc., 1921, 319–327. [Advance copy.]

NITROBENZENE was reduced electrolytically at 30° C. in strong sulphuric acid (90–91%), free from iron and heavy metals, using platinum electrodes, with a cathode current density of 6–8 amp. per 100 sq. cm., and anode density double. Glazed earthenware cells with porous diaphragms of thin, dense porcelain were used. Under these conditions minimum sulphonation was obtained. By washing the cell sludge with commercial hydrochloric acid, centrifuging, and treating with pure hydrochloric acid *p*-aminophenol hydrochloride was obtained, the yield being 40–60% on the nitrobenzene (exclusive of mother liquors which yield a further 10%). Apart from the cost of platinum, the most important economic factor is the necessity of providing for the cheap concentration of the spent acid. Cost of upkeep, owing to the excessive corrosive action of the materials on the apparatus, is also considerable.—P. V. M.

**Phenoarsine; Derivatives of —.** W. L. Lewis, C. D. Lowry, and F. H. Bergeim. J. Amer. Chem. Soc., 1921, 43, 891–896.

6-CHLOROPHENARSIAZINE, m.p. 193° C., has a greater irritant action on the skin and mucous membrane

and is more toxic than diphenylchloroarsine, m.p. 37°–38° C., b.p. 333° C., which in turn is more irritant and toxic than 6-chlorophenoarsine,  $O(C_6H_4)_2AsCl$ , m.p. 124° C., or chlorophenyl-naphthylarsazine, m.p. 228° C. The chlorine in 6-chlorophenoarsine may readily be replaced or displaced, and a number of new compounds have been prepared from it. (Cf. J.C.S., June.)—W. G.

**Chlorovinylchloroarsines.** S. J. Green and T. S. Price. Trans. Chem. Soc., 1921, 119, 448–453.

THREE products are formed when acetylene acts upon anhydrous arsenic trichloride in the presence of anhydrous aluminium chloride, either in the pure state or in solution in an anhydrous solvent, at 15° C.  $\beta$ -Chlorovinylchloroarsine,  $CHCl:CH:AsCl_2$ , colourless, b.p. 93° C. at 26 mm., or 96° C. at 30 mm., possesses a powerful vesicant action.  $\beta\beta'$ -Dichlorodivinylchloroarsine,  $(CHCl:CH)_2AsCl_2$ , b.p. 130°–133° C. at 26 mm., colourless, possesses less powerful vesicant action than the primary compound.  $\beta\beta\beta'$ -Trichlorotrivinylarsine,  $(CHCl:CH)_3As$ , colourless, b.p. 151°–155° C. at 28 mm., m.p. 3°–4° C. All are insoluble in water and dilute acids, but are soluble in organic solvents, with the exception of the tertiary compound which is insoluble in alcohol. Halogens are readily absorbed with the formation of definite characteristic solid compounds. The tendency is for the tertiary derivative to be formed largely at the expense of the others, but it is readily changed to a mixture of the primary and secondary compounds by heating with arsenic trichloride at 200°–250° C.

—P. V. M.

**$\beta\beta'$ -Dichlorodiethyl disulphide.** G. M. Bennett. Trans. Chem. Soc., 1921, 119, 418–425.

$\beta\beta'$ -DICHLORODIETHYL disulphide is synthesised through dihydroxydiethyl disulphide from either monothioethyleneglycol by oxidation with potassium permanganate etc., or from ethylene chlorhydrin and sodium disulphide. Heating with concentrated hydrochloric acid converts the dihydroxy compound into  $\beta\beta'$ -dichlorodiethyl disulphide, b.p. 15° C. at 30 mm., sp. gr. 1.3375 at 20°/4° C. The pure disulphide closely resembles the monosulphide in properties, but has only one-third its vesicant action. Oxidation with nitric acid gives quantitatively  $\beta$ -chloroethanesulphonic acid. In the light of his results the author discusses the condition of the system arising from the interaction of sulphur monochloride and ethylene, and concludes that the liquid produced at temperatures below 35° C. is a two-phase system in which the continuous phase is nearly pure dichlorodiethyl monosulphide and the disperse phase a liquid consisting chiefly of sulphur.

—P. V. M.

**Sulphur monochloride and substituted ethylenes; Interaction of —.** W. J. Pope and J. L. B. Smith. Trans. Chem. Soc., 1921, 119, 396–400. (Cf. Gibson and Pope, J., 1920, 333 A.)

FROM an examination of the interaction of ethylene, propylene, and  $\beta$ -butylene with sulphur monochloride, it is shown that the formation of the corresponding  $\beta$ -chloro-substituted alkyl sulphide and sulphur is general for ethylenic hydrocarbons.  $\beta$ -Dichlorodipropyl sulphide  $(C_3H_5Cl)_2S$  boils at 105°–106° at 11 mm., and  $\beta\beta'$ -dichloro-*sec*-butyl sulphide  $(CH_3CH_2Cl)_2S$ , at 121°–122° at 11 mm. The speed of absorption varies in each case, and of the three substances examined is greatest for  $\beta$ -butylene. Under comparable conditions the speed of absorption, i.e., the rate of interaction, is represented by ethylene:propylene: $\beta$ -butylene = 1:1.33:4. No analogous reaction was obtained between sulphur monochloride and trichloroethylene, the sulphur monochloride acting merely as a chlorinating agent with the formation of pentachloroethane.—P. V. M.



**Mercaptans; Catalytic preparation of** — R. L. Kramer and E. E. Reid. J. Amer. Chem. Soc., 1921, 43, 880—890.

By passing alcohol vapour and hydrogen sulphide in equimolecular proportions at the rate of 1 g.-mol. in 6 hrs. over pumice coated with thorium oxide at 80° C., the following percentage yields of mercaptans were obtained: methyl 41%, ethyl 35%, propyl 45%, butyl 52%, isobutyl 45%, isoamyl 47%. Propyl, butyl, and isoamyl mercaptans give constant boiling mixtures with the corresponding alcohols and ternary mixtures with the alcohols and water. (Cf. J.C.S., June.)—W. G.

**Terolein.** C. Moureu, C. Dufraisse, A. Lepape, P. Robin, J. Pougnet, A. Boutaric, and E. Boismenu. Ann. Chim., 1921, 15, 158—211.

A more detailed account of work already published. Cf. J.; 1920, 42 A, 82 A, 173 A.)—W. G.

**Formaldehyde; Distillation of aqueous solutions of** — J. A. Wilkinson and I. A. Gibson. J. Amer. Chem. Soc., 1921, 43, 695—700.

The distillate from dilute aqueous solutions of formaldehyde (below 8%) is always stronger than the original solution, but with higher concentrations the distillate is always weaker. With low concentrations the residue is always weaker than the last distillate, but with high concentrations the residue is always stronger.—J. F. S.

**Cyanamide; Passage from guanidine to — and from diguanidine to dicyanodiamide.** G. Pellizzari. Atti R. Accad. Lincei, 1921, [V], 30, I, 171—175.

Various reactions similar to the conversion of *o*-phenylenediguanide into  $\beta$ -cyano-*o*-phenyleneguanidine by treatment with nitrous acid (Atti R. Accad. Lincei, 1921, [V], 30, I, 39) have been discovered. Thus, nitrous acid converts diguanide into cyanoguanidine or dicyanodiamide, and guanidine partly into cyanamide. In the latter case, the formation of nitrosoguanidine as an intermediate compound has been detected and it is probable that in the other instances analogous intermediate products are formed. (Cf. J.C.S., June.)—T. H. P.

**Cyanogen bromide and iodide; Preparation of** — V. Grignard and P. Crouzier. Bull. Soc. Chim., 1921, 29, 214—217.

An aqueous solution of sodium cyanide is run slowly on to bromine covered with a small amount of water, with constant stirring, the temperature not being allowed to rise above 25° C., until the colour due to the bromine has disappeared. Chlorine is then bubbled through the liquid and, at the same time, more sodium cyanide is run in, drop by drop, keeping the liberated bromine in slight excess, until the whole of the sodium bromide originally formed is decomposed, when a very slight excess of the cyanide is added. The cyanogen bromide, which crystallises out, may be filtered off, or the contents of the flask may be distilled, the vapour being passed over anhydrous calcium chloride at 80° C. A yield of 91% is obtained. Cyanogen iodide may be prepared in a similar manner, but it is best isolated by extraction with ether. A yield of 89% is obtained.—W. G.

**Essential oil of hamagô.** Y. Shinozaki. Kôgyô-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, 24, 191—202.

By the distillation of the leaves and twigs of *Vitex trifolia*, L. (*Vitex ovata*, Mak.) (Jap., hamagô), grown in the Ogasawara Islands and Kanagawa Prefecture, 0.11—0.28% of an oil having a brown colour and characteristic odour was obtained. Three samples of the oil had the following char-

acters:—Sp. gr. at 15° C., 0.8908—0.9141;  $n_D^{20}$  = 1.4707—1.5010; optical rotation,  $-39^\circ 6'$  to  $-47^\circ 20'$ ; acid value, 0.0—1.75; saponif. value, 23.38—38.70; saponif. value after acetylation, 40.14—53.34. The chief constituents of the oil were *l*- $\alpha$ -pinene, which does not give solid nitrosochloride; camphene (percentage with pinene: 55%); terpinyl acetate (10%), b.p. 84°—86° C. at 15 mm., sp. gr. 0.9629 at 15° C.,  $n_D^{20}$  = 1.4670; a diterpene-alcohol ( $C_{26}H_{42}O$  or  $C_{26}H_{44}O$ ) (20%), b.p. 165°—167° C. at 4 mm., sp. gr. 0.9760 at 15° C.,  $n_D^{20}$  = 1.5143, optical rotation,  $-46^\circ 39'$ , and saponif. value after acetylation 42.02. The oil is separated into its three chief constituents by fractional distillation, and the middle part is suitable for perfumes, as it is mainly composed of terpinyl acetate.—K. K.

**Essential oil of shimamuro.** Y. Shinozaki. Kôgyô-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, 24, 202—208.

By distillation of the leaves and twigs of *Juniperus taxifolia*, Hook. et Arn. (Jap., shimamuro), a coniferous plant, indigenous to the Ogasawara Islands, 0.24% of a light green oil was obtained. Two specimens of the oil had the following characters:—Sp. gr. at 15° C., 0.8675, 0.8701;  $n_D^{20}$  = 1.4702, 1.4718; optical rotation, —,  $-29^\circ$ ; acid value, 0.94, 0.0; saponif. value, 11.69, 10.37; saponif. value after acetylation, 19.60, 18.89. The oil contains  $\alpha$ -pinene (above 50%, mainly *l*- $\alpha$ -pinene containing small quantity of *r*- $\alpha$ -isomer) and possibly a bicyclic terpene, a free alcohol ( $C_{15}H_{26}O$ ), ester, sesquiterpene, and sesquiterpene alcohol.—K. K.

**Narcotic action of light petroleum.** Fühner. See IIa.

**Amygdalinase and amygdalase.** Bertrand and Compton. See XVIII.

#### PATENTS.

**Dichloroethylene; Manufacture of** — A. Wacker, Ges. für Elektrochem. Industrie. E.P. 156,080, 22.10.20. Conv., 23.12.19.

DICHLOROETHYLENE is obtained in 85—88% yield by passing the vapour of tetrachloroethane mixed with steam through a chamber charged with iron, aluminium turnings, or granulated zinc. The crude product issuing from the chambers is condensed and fractionally distilled, the fraction collected between 48° and 60° C. being a mixture of two stereo-isomeric dichloroethylenes. The small higher fraction of unchanged tetrachloroethane is returned to the process.—G. F. M.

**Tubercle bacillus and other germs; Detoxication of — for the preparation of vaccines.** D. Thomson. E.P. 161,341, 23.1.20.

THE germ cultures are separated into a number of non-toxic fractions by successive extractions with either "antiformin" (15% sodium hydroxide plus 15% sodium hypochlorite solution) or *N*/1 sodium hydroxide, *N*/1 hydrochloric acid, absolute alcohol, and chloroform. The insoluble matter is separated after each extraction by centrifuging, and the clear solution is removed for further treatment. The portions extracted by the alkaline and acid solutions are again fractionated by precipitating a portion of the dissolved material with acid and alkali respectively, and subsequently adding absolute alcohol to the liquors to precipitate second fractions. The alcohol extract is precipitated by the addition of normal aqueous sodium chloride, and the final portion extracted with chloroform is recovered by evaporating the solvent. The precipitates are mixed together, with the exception in certain cases of the fractions precipitated by alcohol from the alkali and acid extracts, which possess provocative properties, and are emulsified with normal sodium chloride solution containing 0.5% of phenol,

to yield a vaccine corresponding to ten thousand millions of germs per c.c.—L. A. C.

*Allyl esters of p-aminobenzoic acid.* R. Adams and E. H. Volwiler, Assrs. to The Abbott Laboratories. U.S.P. 1,360,994, 7.12.20. Appl. 10.5.20.

ALLYL esters of amino derivatives of aromatic acids containing a benzene nucleus are prepared by reduction of the corresponding nitro derivatives. The allyl ester of p-nitrobenzoic acid, prepared from p-nitrobenzoyl chloride and allyl alcohol, boils at 178° C. at 19 mm., and on reduction yields the allyl ester of p-aminobenzoic acid, white crystals, m.p. 51°–52° C., of value as an anæsthetic. The dihydrochloride of the allyl ester of 3,5-diaminobenzoic acid is a yellowish solid, m.p. 224° C. (decomp.).

*Phenylcinchoninic acid salt.* M. E. Wolcemp, Assr. to The Abbott Laboratories. U.S.P. 1,361,128, 7.12.20. Appl. 15.9.17.

Acem sodium phenylcinchonate,  $C_{16}H_{11}NO_3Na$ ,  $C_{16}H_{11}NO_3$ , prepared by the action of the calculated quantity of sodium hydroxide solution on the acid and dried below 100° C., forms citron-yellow crystals, freely soluble in water and exhibiting the same medicinal properties as the free acid.

*Acetylation of hydrocarbons and subsequent recovery of the products of the reaction.* G. T. Koch and A. L. Stallkamp, Assrs. to The Ohio Fuel Supply Co. U.S.P. 1,374,668, 12.4.21. Appl. 4.11.20.

CHLOROHYDROCARBONS of the paraffin series are converted into acetates (e.g., amyl acetate) by heating at 250°–450° F. (120°–230° C.) under a pressure of 200–225 lb. per sq. inch with an alkali acetate in presence of activated carbon or "hatchite" as a catalyst. The product is fractionally distilled and the lower fractions returned to a subsequent operation.—G. F. M.

*Organic compounds; Preparation of — by chemical reactions promoted by light.* Farbenfabr. vorm. F. Bayer und Co. G.P. 297,933, 25.6.15.

THE light of the Osram half-watt lamp is used, which is nearly equivalent to sunlight in its chemical effect. Examples are the preparation of xylol chloride from xylene, pinacone from acetone and isopropyl alcohol, etc.—C. I.

*Cyanamide; Preparation of solutions rich in — from calcium cyanamide.* A.-G. für Stickstoffdünger. G.P. 302,515, 5.11.16.

CALCIUM cyanamide is extracted with water and the solution treated with carbon dioxide or absorbing materials, e.g., earth or peat, to remove the lime. The resulting liquor containing 2–3% N is used to extract further quantities of calcium cyanamide. No polymerisation of the cyanamide in solution takes place during the operation.

—A. R. P.

*Alkali cyanamides; Process for preparing —. Preparation of alkali cyanamide solutions.* E. Hene and A. van Haaren. G.P. (A) 306,315, 28.9.16, and (B) 307,011, 17.11.16.

(A) CALCIUM cyanamide and alkali sulphate are allowed to interact at temperatures below 100° C., preferably 25°–35° C., in water. Excess of alkali sulphate reduces the amount of calcium cyanamide left in solution, but a double sulphate is then formed. (B) Instead of alkali sulphate a double alkali-calcium sulphate may be used. To a weak solution of alkali cyanamide, calcium cyanamide and sufficient alkali sulphate to form mono-alkali cyanamide along with a double sulphate are added. The resulting mud is used with more calcium cyanamide and water to form fresh weak solution,

and the mono-alkali cyanamide which still contains calcium is treated with fresh alkali sulphate.

—C. I.

*Fatty acids and aldehydes; Process for the preparation of —.* C. Harries, R. Koetschau, and E. Alhrecht. G.P. 332,594, 23.3.17. Addn. to 324,663 (J., 1920, 765 A).

OZONIDES prepared from petroleum, shale oil, or tar oils or the compounds formed by their reaction with water vapour or alkali are treated with oxidising agents, e.g., hydrogen peroxide and alkali, Caro's acid, halogens, chromic acid, glacial acetic acid and permanganate.—C. I.

*5(4)-Aralkylaminoalkyliminazoles; Manufacture of —.* O. Gerngross. G.P. 332,955, 10.6.14. Addn. to 278,884 (J., 1915, 250).

COMPOUNDS of the general formula, iminazoly.  $(CH_2)_x.NH.(CH_2)_y.Ar$ , are prepared by the interaction of iminazolyalkyl chlorides and aralkylamines or aralkyl chlorides,  $Ar.(CH_2)_x.Cl$  and iminazolealkylamines, or the salts of the compounds respectively. By interaction of 4(5)-methyl-5(4)-chloromethyliminazole hydrochloride and  $\beta$ -phenylethylamine, a mixture of secondary 4(5)-methyl-5(4)-phenylethylaminomethyliminazole and tertiary di-5(4)-methyliminazoly-4(5)-methyl- $\beta$ -phenylethylamine is obtained. They are separated by fractional crystallisation of their picrates from acetone, the picrate of the tertiary base being more soluble. By heating  $\beta$ -p-hydroxyphenylethyl chloride with  $\beta$ -4(5)-iminazolyethylamine in dry methyl alcohol for 12 hrs. at 100° C. under pressure, 4(5)-4'-hydroxyphenylethylaminomethyliminazole,  $OH.C_6H_4.(CH_2)_2.NH.(CH_2)_2.C_6H_4.N$ , is formed. It melts at 156° C. and is slightly soluble in water. The compounds have a physiological effect similar to that of ergot.—C. A. C.

*Formic acid esters; Preparation of —.* Elektrochem. Werke C.m.b.H., H. Bosshard, and D. Strauss. G.P. 334,298, 19.11.16.

FORMIC acid and alcohols of the highest concentration practicable are caused to react in the theoretical proportions in presence of calcium chloride without addition of mineral acids. The apparatus may be made of iron, and the calcium chloride recovered.—C. I.

*Hexamethylenetetramine; Preparation of addition products of —.* J. D. Riedel A.-G. G.P. 334,709, 31.12.18.

IF a mixture of hexamethylenetetramine and monohalogen-acetic acid, in an anhydrous solvent, is heated, the salt first formed is converted into a substance resembling betaine hydrochloride. The presence of a little water or the use of halogenated higher fatty acids leads to the formation of hexamethylenetetramine-halogen hydrate. The addition products from monochloro- and monobromoacetic acids are coarsely crystalline substances, easily soluble in water, slightly in alcohol, and insoluble in acetone and ether. On crystallisation from aqueous alcohol the halogen hydrate compound is formed. The aqueous solution, which tastes acid, evolves formaldehyde on heating. The substances are strong bactericides.—B. V. S.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Photographic plate; Extinction by a blackened — as function of wave-length, quantity of silver, and size of the grains.* A. Deumens. Proc. Roy. Acad. Amsterdam, 1921, 23, 848–865.

Two series of plates were prepared, one set developed with varying times, or temperature, or developer strength, in a caustic quinol developer

and the other set with similar variations in a glycin developer. All the plates were examined as to density, amount of silver, and grain size. The density value was found to vary with the wavelength of the light by which it was measured to an extent which varied with the conditions of development, this being one of the factors determining grain size. Plates developed in a normal glycin developer were practically neutral in colour in the yellow-green portion of the spectrum and suitable therefore for use as reducers of light intensity in photometric measurements etc. using yellow-green light. The relation between quantity of silver and density is shown to vary with grain size. It is suggested that the proportionality (photometric constant) previously found by Hurter and Driffeld, Sheppard and Mees, and Eder was due to their use of silver images with grains of equal size. Some figures are also given for collargol.—B. V. S.

[Photographic] red sensitiveness of silver iodide. R. B. Archey. *Phot. J.*, 1921, 61, 235–236.

In using various cinematograph positive films a considerable variation in the case of fogging by the dark-room light was observed, both between different brands of film and between different emulsions of the same manufacture. Film was exposed 30 mins. to a 25 c.p. lamp behind a Wratten No. 1 safelight at a distance of 1 ft., and developed under constant, normal conditions. The resulting density measured on a König-Martens photometer was found to be proportional, not to the speed of the plate, but to the contrast. In one case the same film was tested at intervals of 1 month for 9 months, the H. and D. speed, developing factor, and red sensitiveness being determined; changes in the two latter corresponded but not with changes in speed. The addition of excess iodide in the manufacture of an emulsion in one case led to a fog value of 2.72. Spectrograms of two films are given at either end of the spectrum in each case, the two spectra at the blue end being practically indistinguishable, while at the red end one film shows no image and the other a very strong one.—B. V. S.

#### PATENTS.

[Photographic] toning-baths for silver-pictures, with tellurium or selenium as the toning agent. Kraft und Steudel, Fabr. phot. Papiere, G.m.b.H. G.P. 334,172, 1.6.20.

In the preparation of toning baths containing a selenium or tellurium salt as toning agent, other known toning agents such as thionrea, hydrazine, or hydroxylamine may be added, along with a salt such as a borate or phosphate, or preferably, acetate, citrate, tartrate, etc., which prevents precipitation of the selenium or tellurium. A bath prepared with tellurous acid and an alkali, such as lithium hydroxide, is preferably acidified. The stability of a toning bath may be increased by the addition of a protective colloid such as albumin or starch. The developed, fixed, and washed print is toned for 3–15 mins. according to the tone required; chloride prints tone more quickly and require a weaker bath. Selenium gives redder tones than tellurium.—B. V. S.

Coloured [photographic] pictures; Process for making —. A. Traube. E.P. 147,103, 7.7.20. Conv., 3.12.18. Addn. to 147,005.

Basic dyes of the thiobenzoyl, thiazine, pyronine, safranin, oxazine, and acridine classes give better results than the dyes mentioned in the chief patent (J., 1921, 325 A), by reason of their greater permanence in the toning processes, combined with easy staining and sufficient colour intensity.—B. V. S.

Photographic reproduction; Process of —. R. John, Assr. to Iconochrome Co. of America, Inc. U.S.P. 1,374,853, 12.4.21. Appl., 25.7.19.

For the production of a transfer printing plate an emulsion is used containing in addition to the light-sensitive material a substance which reacts with the product of light action on the sensitive material to form a hardening agent, and a further substance having a neutralising effect on the hardening agent which is added in sufficient proportion to make the amount of hardening proportional to the amount of image.—B. V. S.

Photosensitising dye of the isocyanine type. Photosensitising dye effective for infra-red radiation. E. Q. Adams and H. L. Haller. U.S.P. (A) 1,374,871 and (B) 1,374,872, 12.4.21. Appl., 28.5 and 17.7.20.

(A) PHOTOSENSITISING dyes are produced by the action of strong bases in alcoholic solution upon the alkyl halides of lepidine. (B) The dyes obtained from the higher alkyl halides of lepidine give sensitiveness in the infra-red. (Cf. J., 1921, 75 A.) —B. V. S.

#### XXII.—EXPLOSIVES; MATCHES.

Corn [maize] cob cellulose; Possible uses of — in the explosives industry. L. G. Marsh. *J. Ind. Eng. Chem.*, 1921, 13, 296–298.

CELLULOSE extracted from maize cobs by treatment with 1% sodium hydroxide, and then 4% sulphuric acid, at 100° C., absorbs 150% of its own weight of nitroglycerin, and is therefore a suitable ingredient for dynamite. In preliminary tests of its absorptive capacity for water, with a view to employing it as a substitute for cotton in nitrocellulose manufacture, samples were totally submerged in less than 12 secs. after dropping on the surface of the water. Some difficulty was experienced in separating nitrated maize cob cellulose from the acid on account of its fineness; screening was ineffective, and it was finally separated by gravity, a mat of nitrated cotton being used. Washing and settling were carried out satisfactorily; pulping was unnecessary, but the usual purification system could be adopted. A yield of about 130% was obtained. The product gave a satisfactory heat test. The nitrogen content was only 12.3%, due to a high proportion of material insoluble in acetone; after removing this, the nitrogen figure was increased to 12.7%. Even by nitrating equal parts of cotton and maize cob cellulose, no material reduction was effected in the acetone-insoluble residue; its structure, consisting of fine, hard grains, militates against the use of maize cob cellulose for the manufacture of perforated powders.—W. J. W.

Trinitrotoluene poisoning; Nature, diagnosis, and prevention of —. C. Voegtlin, C. W. Hooper, and J. M. Johnson. Hygienic Lab., U.S. Public Health Service. Bull. 126, 1920, 7–181.

NUMEROUS tests were made on dogs, which are more sensitive than other animals, and develop the typical symptoms noted with human beings. The toxic action of TNT is essentially due to 2,4,6-trinitrotoluene, and not to impurities. As indicated by Moore (Causation and Prevention of Trinitrotoluene Poisoning, Medical Research Committee, London, 1917), TNT is not excreted as such, but is converted after absorption into a hydroxylamine compound. This compound may also undergo reduction to the mono- or diamino-derivative which do not give the Webster reaction but have the same pharmacological action as TNT. Differences in resistance of different individuals may be due to the fact that more resistant animals oxidise the methyl

group of TNT more readily. Reliance on clinical symptoms alone in the diagnosis of TNT poisoning in munition workers is not sufficient, as marked blood changes may be present, even when the workers do not exhibit signs of cyanosis, pallor, or icterus; an examination of the blood is therefore essential. Sex has no influence on susceptibility to poisoning, and individual susceptibility, as with dogs, explains the absence of relation between time of exposure to the poison and contraction of anaemia. The authors do not concur with Moore's view that all TNT poisoning is caused by skin absorption, and that individual susceptibility is accounted for by difference in skin permeability. Preventive measures should include efficient ventilation and the use of induced draught for cleaning floors, etc. Overalls extending over the ankles and a head-dress should be worn; gloves are useless. A skin wash of 10% sodium sulphite solution has proved satisfactory. Workers' diet should include a fair proportion of meat, vegetables, bread, and fruit. Although to a certain extent intermittent employment reduces the health risk, it does not entirely prevent poisoning, as the system retains TNT for a long time. Workers should undergo a preliminary medical examination, and none suffering from anaemia or liver disease should be employed.

—W. J. W.

**Parazol (crude dichlorodinitrobenzene); Toxicity of** — C. Voegtlin, A. E. Livingston, and C. W. Hooper. Hygienic Lab., U.S. Public Health Service. Bull. 126, 1920, 183—202.

**PARAZOL**, which was extensively used as a high explosive during the war, is a mixture of three isomers: *m*-dinitro-*p*-dichlorobenzene, *o*-dinitro-*p*-dichlorobenzene, and *p*-dinitro-*p*-dichlorobenzene; in addition, a small fraction consisting of *p*-nitrochlorobenzene has been separated from it. The action on the skin of crude and recrystallised parazol, as well as of two of its isomers and of *p*-nitrochlorobenzene, was studied with rabbits, and confirmed in some cases with human skin. In each case dermatitis was produced. More severe lesions were caused by the crude product than by its isomers, and of these latter the *m*-dinitro compound proved more injurious than either the *o*-dinitro compound or *p*-nitrochlorobenzene. In regard to the action on the eyes, no effect was produced when the products were applied as a 1% solution in olive oil; on the other hand, the dry powder caused marked conjunctivitis; that this result was not due merely to mechanical irritation was proved by the varying effect produced by the different products, crude parazol being the most active. Crude parazol when injected subcutaneously caused extensive edema and induration, sometimes leading to the formation of an abscess. When introduced through the mouth it was absorbed without gastro-intestinal irritation. Relatively large amounts of parazol are required to produce anaemia, and it may therefore be regarded as a low grade poison. The active agents in causing anaemia are probably the nitro-groups, which, as with TNT, become reduced in the body. On the other hand, dermatitis and conjunctivitis depend on the presence of chlorine in the molecule. To protect parazol workers contamination of the air with the material should be prevented, and goggles should be worn. The provision of proper overalls is essential; skin varnishes are unreliable.—W. J. W.

**Mercury fulminate; Skin irritation and systemic effects produced by** — A. E. Livingston. Hygienic Lab., U.S. Public Health Service. Bull. 126, 1920, 203—211.

APPLICATION of mercury fulminate to the shaved skin of rabbits invariably caused a marked lesion after 2—3 hrs., and blanching of the skin took

place, followed by edema; after a longer period a hemorrhagic condition replaced the blanching. Applications of fulminate to the human skin produced no effect whatever, but no conclusion can be drawn from this as to its effects on workers in factories, where friction, lack of cleanliness, etc., may cause the poison to enter the skin, and where individual susceptibility has an important bearing. In regard to the systemic effects of mercury fulminate, there is no evidence of fatal results brought about by absorption through the skin, nor could it be detected in the urine, after skin application. When administered as capsules through the mouth the minimum lethal dose for rabbits appears to be about 20 mg. per kg. As a preventive measure against fulminate poisoning, a shellac skin varnish has given good results. Sodium thiosulphate solution is beneficial if used as a wash; it is, however, useless for treatment after injury.—W. J. W.

**Detonators and percussion caps; Examination of** — A. Langhans. Z. ges. Schiess- u. Sprengstoffw., 1921, 16, 49—52, 57—59.

MICROSCOPICAL examination of cap and detonator compositions affords a means of identifying their components. In a quantitative examination the risk involved in removing the composition from its metal container is avoided by submitting the whole detonator or cap to solution and analysis. The author describes a method of analysis. The composition of the caps was as follows: Mercury fulminate, 0.051 g. (mercury, 0.038 g.); antimony sulphide, 0.208 g.; potassium chlorate, 0.339 g.; powdered glass, 0.013 g.; potassium nitrate, 0.0684 g.; sulphur, 0.0081 g.; carbon, 0.0135 g.; copper, 0.79 g. Four caps were treated for 2 hrs. with a mixture of 50 c.c. of nitric acid and 25 c.c. of water. The solution was evaporated to dryness, and the residue boiled three times with dilute nitric acid, and filtered; powdered glass, together with some charcoal and sulphur, remained on the filter. After dilution of the filtrate to 500 c.c., 100 c.c. was treated with hydrogen sulphide; the precipitate was treated with sodium sulphide solution and potassium hydroxide, and the solution filtered from the undissolved copper sulphide, which was dissolved in acid and electrolysed. The filtrate containing sulphides of antimony and mercury in solution was acidified, and the precipitated sulphides filtered off and treated with ammonium sulphide for a prolonged period; the antimony sulphide was precipitated with sulphuric acid, whilst the undissolved mercury sulphide was dissolved in nitric acid and electrolysed. For the estimation of potassium chlorate and nitrate the filtrate from the original hydrogen sulphide precipitation was evaporated to expel hydrogen sulphide, and the residue then treated with water and sulphuric acid and again evaporated to dryness. From the potassium sulphate found the respective amounts of chlorate and nitrate were calculated. For the analysis of detonators an analogous method to the above may be employed. For mercury fulminate and potassium chlorate compositions a method of estimating chlorate by the use of nitron is also applicable.—W. J. W.

#### PATENTS.

**Explosives [for detonators].** A. G. Lowndes. E.P. 160,953, 6.1.20.

THE sensitiveness of explosives, such as lead azide, is said to be due more to the structure and cleavage than to the size of the crystals. By adding a non-colloidal solution, which has no action on lead azide, to the liquid in which it is precipitated so as to increase the density of the liquid, the azide may be obtained in the form of anhedra, flakes, or thin plates, of less sensitiveness than the holohedra usually formed. Thus, 200 c.c. of sodium azide solution (40 g. per l.) is added to a mixture of

200 c.c. of lead acetate solution (117 g. per l.) and 1000 c.c. of sodium nitrate solution (450 g. per l.), all at 15° C. Alternatively, equivalent solutions of sodium azide and lead acetate may be run into sodium nitrate solution. (*Cf.* E.P. 142,898; J., 1920, 529 A.)—W. J. W.

*Explosive; High — and process of making same.* W. R. Swint, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,373,844, 5.4.21. Appl., 6.5.18.

AN explosive composition consists of wet nitro-cellulose of a high degree of nitration and a liquid nitro-derivative of a hydrocarbon.—W. J. W.

*Explosive.* G. Weber, Assr. to Soc. Les Petits Fils De Francois De Wendel et Cie. U.S.P. 1,375,243, 19.4.21. Appl., 27.9.19.

AN explosive contains a combustible organic substance adapted to absorb liquid air, and blast-furnace dust.—L. A. C.

*Detonators; Process for destroying waste — and recovering copper and mercury from the same.* W. Eschbach. G.P. 298,860, 29.11.16.

THE detonators are placed in a rotary drum, provided with a suitable shock-absorbing device, and treated with a hot solution of sodium carbonate, which loosens the composition by dissolving its potassium chlorate or perchlorate, and converts the mercury fulminate into mercuric carbonate and sodium fulminate. After filtering and washing this mixture the mercury is recovered from it electrolytically. The empty shells are washed and introduced in small quantities into a furnace in which any remaining traces of explosive are decomposed.—W. J. W.

*Mixed acid; Process for preparing —.* Chem. Fabr. Griesheim-Elektron. G.P. 303,892, 1.3.16.

DILUTE nitric acid is absorbed, *e.g.*, in a tower, by a counter current of cooled concentrated sulphuric acid.—C. I.

*Ammonium nitrate and carbon; Process of obtaining mixtures of — which can be cast.* Verein Chem. Fabr. in Mannheim. G.P. 309,288, 5.9.17.

WOOD charcoal of a definite, not too low adsorptive power and water content, is mixed with ammonium nitrate. The melting point of the mixture is lowered by the addition of carbon or oxygen compounds; *e.g.*, 15 pts. of finely powdered wood charcoal is saturated with 25% of its weight of water, then mixed with 77 pts. of ammonium nitrate and 8 pts. of sodium nitrate, the mixture heated to 110° C., and cast in the desired shape.—C. I.

### XXIII.—ANALYSIS.

*Viscosimeter.* R. Fischer. Z. angew. Chem., 1921, 34, 153—154.

THE tube of a falling-sphere viscosimeter described previously by the author (*Chem.-Zeit.*, 1920, 44, 622) may be widened so that the path of the sphere is not affected by the tubulus for the thermometer; the apparatus may be standardised with different sized spheres for use with liquids of low and high viscosities, and, when dealing with aqueous liquids, the electrical contacts at the bottom of the tube may be covered with a layer of carbon tetrachloride which is introduced before the liquid.—W. P. S.

*Combustion analysis by means of tellurium dioxide.* T. R. Glauser. Z. angew. Chem., 1921, 34, 154—155, 157—159, 162—163.

A RECORD of further work on a method described previously (J., 1914, 223). Whilst the carbon in many substances (*e.g.*, iron, heavy metals, graphite) is oxidised readily to carbon dioxide by heating with tellurium dioxide, the reaction proceeds very

slowly in the case of ferrosilicon, carborundum, etc., and for the determination of carbon in these it is better to use lead oxide or a mixture of lead oxide and tellurium dioxide as the oxidising agent.

—W. P. S.

*Refractive indices of liquids; Simple instrument for the determination of the —.* W. A. M. Smart and F. A. Hocking. Pharm. J., 1921, 106, 286—288.

A VERTICAL rod, fixed to a wooden base, carries a sliding device to which is attached an observation lens; a piece of hat-pin is also attached to the sliding device below the lens so that its end is vertically below the centre of the lens. A thin mirror is let into the base and a convex lens of +61 to +10D is laid on the mirror. The slider is raised or lowered until the pin and its image (as seen by reflection from the mirror and through the lens) are exactly coincident. The distance between the pin and the centre of the lens is then read off on a scale fixed to the vertical rod. A few drops of water are then placed between the mirror and the lens and the new focal position read off after adjusting the slider. This operation is repeated, using the liquid the refractive index of which is to be determined in place of the water. The refractive index,  $\mu_2$ , of the liquid is then calculated by the formula,  $\mu_2 = \mu(f - F_1)F_1 + (F_2 - F)f/F_2(f - F_1)$ , where  $\mu$  is the refractive index of water,  $f$  the focal length of the lens when air is between it and the mirror,  $F_1$  the focal length of the water-lens combination, and  $F_2$  that of the liquid-lens combination, these lengths being read in mm. on the scale.—W. P. S.

*Enamelled bombs; Use of — in calorimetry.* C. Matignon and G. Marchal. Comptes rend., 1921, 172, 921—922.

ENAMELLED bombs of recent manufacture are likely to be attacked by the dilute nitric acid, such as is always formed under the ordinary conditions of calorimetric work. To obviate this source of error new bombs should be filled with N/1 nitric acid and submitted to its action for 4—5 hrs. before being used.—W. G.

*Filtration in dry or indifferent gases; Apparatus for —.* A. Wolfram. Ber., 1920, 54, 857—859.

THE apparatus permits the preparation, filtration, and desiccation of a hygroscopic substance in a single operation, with complete exclusion of moisture. The reaction vessel is a round-bottomed flask provided with a tubulus in one side by means of which a dropping funnel is attached, into which a dried gas can be passed as required. The accurately-ground neck of the flask presses a hardened filter paper against a perforated porcelain disc placed in a filter tube similar to those used with Gooch crucibles, the junction between tube and flask being made by a rubber ring. The narrow end of the filter tube is connected by a two-holed rubber stopper with a separating funnel, the other hole permitting attachment to an exhaust pump. The complete apparatus is mounted in a slightly inclined position and the reacting substances are mixed in the flask; it is subsequently brought into a vertical position, whereby the mixture flows on to the filter paper and the precipitate is filtered by suction. The filtrate is run off through the larger funnel and the precipitate is washed with liquid introduced through the smaller one. It is finally desiccated by aspirating a current of dry air or other suitable gas through it.—H. W.

*Spot reactions in qualitative analysis.* E. Hauser. Z. anal. Chem., 1921, 60, 88—89.

THE advantages of spot reactions on filter paper for the identification of simple substances or of mixtures, as described recently by Feigl and Stern (J., 1921, 280 A), are confirmed.—W. P. S.

*Metals of the second group; Separation of —.* G. G. Longinescu and G. P. Theodorescu. Bul. Sci. Acad. Roumaine, 1920, 6, 159—164.

THE mixed sulphides are heated with ammonium carbonate solution, and filtered; the arsenic alone is dissolved. The precipitate is then dissolved in hydrochloric acid with the addition of potassium chlorate, the solution diluted, cooled, and filtered to remove the greater part of the lead chloride. The filtrate is neutralised with sodium carbonate, sodium hydroxide is added, the mixture boiled, and filtered. Mercury, bismuth, copper, and cadmium remain in the insoluble precipitate, whilst tin, antimony, and the remainder of the lead pass into the filtrate. The precipitate is dissolved in hydrochloric acid and the solution rendered ammoniacal; mercury and bismuth are precipitated and these two metals are then separated and identified by the usual methods and tests, as are also the lead, tin, and antimony.—W. P. S.

*Mercury; Identification of — as cuprous mercuric iodide.* P. Artmann. Z. anal. Chem., 1921, 60, 81—88.

WHEN precipitated cuprous iodide is treated with a drop of a mercury salt solution, a bright red coloration is obtained. The test may be made on a microscope slide, or better, on filter-paper, the cuprous iodide being applied to the latter and dried at a low temperature before the mercury solution is added. The reaction may be obtained with a drop of solution containing 2 mg. of mercury per litre. The acid concentration of the mercury solution must not be greater than  $N/10$ , and bismuth salts and substances which are reduced readily must not be present.—W. P. S.

*Iron; Determination of — by the cupferron method.* G. E. F. Lundell. J. Amer. Chem. Soc., 1921, 43, 847—851.

PRECIPITATION of iron by cupferron is quantitative in hydrochloric and sulphuric acid solutions containing as much as 20% by volume of either acid. The precipitate is not dissolved by washing with cold dilute hydrochloric acid (1:9). Ammoniacal wash waters, which need rarely be employed, may cause losses. These are always indicated by the formation of turbid filtrates. Perfectly clear filtrates and washings are absolutely essential in accurate determinations of iron by the cupferron method. The precipitates occasionally exhibit a tendency to creep through the filter, consequently when the filtrate or wash water is even opalescent the need for such corrective treatments as re-filtration or digestion in the cold followed by re-filtration is indicated.—J. F. S.

*Electrometric titration; Determination of iodic acid and silver by —.* W. S. Hendrixson. J. Amer. Chem. Soc., 1921, 43, 858—866.

IODATE may be accurately determined by reducing it with a known excess of iodide in dilute sulphuric acid and titrating the excess with permanganate by the electrometric method previously described (J., 1921, 241 A). Iodides can be titrated directly with iodate in dilute sulphuric acid solution by the electrometric method. Hydrochloric acid may not be substituted for sulphuric acid except within very narrow limits of concentration in the determination of iodide with either permanganate or iodate. These estimations may be made in the presence of chloride not exceeding about  $0.1N$  or in the presence of nitric acid of higher concentrations if it contains only traces of nitrous acid. Silver may be accurately determined electrometrically with pure iodide and permanganate solution. Pure silver may be used as a standard in determining the values of both iodide and permanganate.—J. F. S.

*Copper; Use of silver cathodes in the electro-deposition of —.* H. W. Doughty and B. Freeman. J. Amer. Chem. Soc., 1921, 43, 700—704.

SILVER cathodes for use in the electro-deposition of copper are cleaned by placing them in contact with pure tin-foil in a boiling 10% sodium hydroxide solution until bright. They are then washed successively with dilute hydrochloric acid, dilute ammonia, and distilled water, rinsed with alcohol, and ignited to burn off the alcohol. The electrolysis is carried out as usual except that the electrode is not immersed in the solution until the current has been switched on. To remove the copper deposit, after the final weighing, the cathode is placed in a wide-mouthed stoppered bottle filled with a 10% solution of trichloro-acetic acid in aqueous ammonia (sp. gr. 0.90) to which an equal volume of water has been added. The copper is removed in 10 mins. and the silver electrode is not attacked. Chloroform or carbon tetrachloride may be substituted for trichloro-acetic acid, but in this case the ammonia solution is made from 1 pt. of ammonia of sp. gr. 0.90 and 1 pt. of alcohol. The reaction is slower and care must be taken that no sulphur compounds are present in the carbon tetrachloride. (Cf. J.C.S., June.)—J. F. S.

*Gold; Electrolytic determination of — and its separation from copper, palladium, and platinum.* W. D. Treadwell. Helv. Chim. Acta, 1921, 4, 364—374.

GOLD may be electrolytically deposited, rapidly and quantitatively, from solutions of the chloride containing acetate, this method admitting of the separation of gold from copper, palladium, and platinum. The readiness with which palladium is attacked in comparison with platinum, when subjected to anodic polarisation in hydrochloric acid solution, serves as a means for the approximate estimation of the palladium content of a precipitate composed of a large proportion of palladium and of little platinum.

—T. H. P.

*Hydrochloric acid; Detection of — in the presence of hydrobromic and hydriodic acids.* G. G. Longinescu and G. Chaborski. Bul. Sci. Acad. Roumaine, 1920, 6, 122—124.

THE mixed salts are heated in a test-tube with a mixture of ethyl alcohol and concentrated sulphuric acid, an inverted funnel containing paper moistened with silver nitrate being placed over the mouth of the test-tube. The hydrochloric acid evolved together with a small amount of iodine is absorbed by the silver nitrate, and when the silver chloride is dissolved subsequently in ammonia the iodine remains insoluble. The hydrobromic acid is decomposed partially and bromoethanes are formed. If the heating is continued after all the hydrochloric acid has been expelled, the bromoethanes and iodine are evolved and may be collected by inverting a moistened beaker over the mouth of the test-tube. The beaker is then washed out with water and the solution treated with chlorine-water after the addition of carbon bisulphide; when sufficient chlorine has been added the violet coloration due to the iodine disappears and the brown coloration of the bromine remains.—W. P. S.

*Nitric acid; Detection of —.* G. G. Longinescu and G. Chaborski. Bul. Sci. Acad. Roumaine, 1920, 6, 176—178.

WHEN a nitrate is heated with a small quantity of benzene and concentrated sulphuric acid, nitrobenzene is formed, and this may be detected readily by its characteristic odour. If bromides or iodides are also present, the odour of nitrobenzene cannot be noticed until after the mixture has been neutralised with sodium hydroxide.—W. P. S.



See also pages (A) 377, *Tar etc. in producer gas* (Jenkner); *Density of petroleum residues* (Predescu). 382, *Sulphite liquors* (Sieber); *Sulphite pulp* (Sieber). 387, *Fluorides* (Garcia); *Hypochlorous acid* (Treadwell). 392, *Sulphur in steel* (Rooney). 396, *Acetyl value* (André); *Oxidised fatty acids* (Goldschmidt and Weiss). 397, *Beeswax* (Jolles). 398, *Turpentine oil* (Marcusson). 401, *Bacteriological analysis of soil* (Wyant). 402, *Artificial honey* (Stanek and Vondrak). 406, *Silage crops* (Neidig and Snyder). 407, *Hydrogen-ion concentration of water* (Kolthoff). 408, *Caffeine in yerba maté etc.* (Ugarte). 409, *Alkaloids in cacao* (Ceriotti); *Alkaloids* (Heiduechka and Wolf); *Hydrocyanic acid* (Denigès). 414, *Detonators* (Langhans).

## PATENTS.

*Gas; [Laboratory] apparatus for the generation of — formed by the action of liquids on solid reagents.* F. Hirsch. E.P. 161,681, 14.1.20.

THE apparatus consists of a reservoir, containing the liquid reagent, connected through a tap with a long tapering generating tower with a perforated bottom constricted to a tube forming a tight connexion with a receiver placed beneath it, into which the spent liquor from the generating tower flows. The receiver is furnished with a gas outlet at the top and a spent liquor outlet at the base. The gas is thereby caused to pass through the mass of the solid reagent in the tower in order to reach the gas outlet, and when the latter and the reservoir tap are closed the further generation of gas ceases.

—G. F. M.

*Gas analysing apparatus.* Svenska Aktiebolaget Mono. E.P. 138,355, 28.1.20. Conv., 29.1.19.

IN gas analysing apparatus operated as described in E.P. 16,561 of 1908 and provided with two or more measuring vessels, synchronous analyses may be carried out by arranging that the gas from only one of the measuring vessels passes through the remaining members of the apparatus, whilst the gas from the other measuring vessels escapes to the atmosphere.—J. S. G. T.

*Analysing gases; Apparatus for —.* R. H. Davis and C. Rosling. E.P. 160,930, 30.12.19.

APPARATUS for analysing gases comprises a vessel containing an absorbent of the constituent to be determined, both ends of the vessel being in the form of diaphragms such as are employed in aneroid barometers. Gas is admitted to the vessel through a valved inlet and the change of pressure due to absorption of a constituent of the gas is transmitted by one of the diaphragms to a pointer moving over a scale. The second diaphragm controls the entry and exit of gas before and after analysis, and serves to retain the absorbent liquid for subsequent analyses.—J. S. G. T.

*Gas-analysing apparatus.* O. Rohde, and Svenska Aktiebolaget Mono. E.P. 160,854, 7.8.19.

IN apparatus in which two measuring vessels are employed for measuring the volume before and after absorption of various constituents, the first measuring vessel is cooled by liquid flowing into its interior, and the second by liquid flowing in a helical coil surrounding the vessel. The liquid is kept in circulation by means of a pump, and preferably a regulating vessel is inserted in the discharge pipe so that the flow of cooling water may be adjusted to about 3 to 5 drops per sec.—J. S. G. T.

*Gas-analysing apparatus.* O. Rohde, Assr. to Svenska Aktiebolaget Mono. U.S.P. 1,373,264, 29.3.21. Appl., 8.9.19.

SEE E.P. 160,854 of 1919; preceding.

*Gas; Apparatus for analysing —.* O. Rohde, Assr. to Aktiebolaget Ingeniörsfirma F. Egnell. U.S.P. 1,374,391, 12.4.21. Appl., 29.1.17.  
SEE E.P. 104,160 of 1916; J., 1918, 447 A.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

## I.—GENERAL; PLANT; MACHINERY.

## APPLICATIONS.

Caldwell. Crushing and grinding apparatus. 13,272. May 10.  
Collins. Furnaces for steam-generators, kilns, etc. 13,842. May 17.  
Cross. Compositions for preventing corrosion and removing incrustation in boilers etc. 13,636. May 13.  
Ingersoll-Rand Co. Evaporising and cooling. 13,814. May 14. (U.S., 5.6.20.)  
Lloyd and Miller. Grinding or crushing machines. 13,564. May 13.  
Lodge Fume Co., and others. 14,230-1. See XI.  
Nitrogen Corp. Catalyst, and method of preparing same. 13,161. May 9. (U.S., 8.5.20.)  
Rigby. Heating or cooling liquids or admixed solids in evaporative etc. treatment. 13,206. May 10.  
Techno-Chemical Laboratories, Ltd., and Boberg. Refrigeration. 13,253. May 10.  
Wade (Union Trust Co.). Regenerative furnaces. 14,152. May 20.  
Weddingen. Removing incrustation from boilers and softening feed-water therefor. 14,147. May 20.

## COMPLETE SPECIFICATIONS ACCEPTED.

17,331 (1917). Krause. Evaporising or cooling solutions, emulsions, and suspensions, and production of chemical reactions. (162,678.) May 19.  
23,244 (1919). Hall. Grinding, crushing, or disintegrating machines. (163,064.) May 25.  
29,488 (1919). Cathcart. Method and apparatus for drying. (162,705.) May 19.  
31,696 (1919). Soc. l'Air Liquide. Separation of constituents of gaseous mixtures. (136,837.) May 19.  
32,639 (1919). Mitchell. Apparatus for making emulsions. (162,719.) May 19.  
3212 (1920). Poore. See II.  
4065 (1920). Engelke. Filtration of liquids. (163,105.) May 25.  
4267 (1920). Merz. Receptacles for use in connexion with the crystallisation of solutions. (163,116.) May 25.  
4729 (1920). Duparc and Urfer. Production of catalysts. (140,061.) May 25.  
7468 (1920). Bonsignori. Evaporator. (162,877.) May 19.  
7713 (1920). Vermaes, and Syndicaat Electrostaal. Rotary kilns. (163,175.) May 25.  
8485 (1920). Larsen. Burning watery materials in kilns. (144,255.) May 25.  
10,947 (1920). Losey. Apparatus for disintegrating fused materials. (162,909.) May 19.  
19,579 (1920). Martini u. Hüneke Maschinenbau A.-G. Protecting inflammable liquids from fire. (147,622.) May 19.  
20,626 (1920). Danhardt. Metallic filter for separating solids from gases. (148,795.) May 19.  
26,941 (1920). Wettig, and Topf u. Söhne. Apparatus for drying granular material. (163,244.) May 25.



## II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### APPLICATIONS.

- Atkinson. Combustion of fuel. 13,323. May 11.  
 Bergh. Retorts for distilling shale etc. 14,153. May 20.  
 Boardman, Madden, West, and West's Gas Improvement Co., Ltd. Destructive distillation of coal etc. 13,633. May 13.  
 Booser. Materials for purifying acetylene. 13,180. May 9.  
 Dunstan and Thole. Treatment of petroleum etc. 13,174. May 9.  
 Dunstan. Refining liquid hydrocarbons. 13,515. May 12.  
 Fournier. Manufacture of artificial fuel. 13,246. May 10. (Fr., 30.7.20.)  
 Kirke. Water-gas plants. 13,637. May 13.  
 Kirke. Combustion of fuel. 13,638. May 13.  
 Meikle. Vertical retort. 13,223. May 10.  
 Nitrogen Corp. 13,813. See VII.  
 Tarrant, and Tarrant, Ltd. Fuel blocks. 13,416. May 11.  
 Torfverwertungsges. Treatment of raw peat. 13,277-8. May 10. (Ger., 22.6 and 6.10.20.)  
 Tulloch. Fuel, and production of gas therefrom. 13,508. May 12.

### COMPLETE SPECIFICATIONS ACCEPTED.

- 20,598 and 25,103 (1919). Low Temperature Carbonisation, Ltd., Davidson, and Armstrong. Distillation of coal etc. (162,684.) May 19.  
 2057 (1920). Goldschmidt and Migeon. See XVIII.  
 2319 (1920). American Coke and Chemical Co. Regenerative coke-ovens etc. (138,125.) May 19.  
 3212 (1920). Poore. Apparatus for carbonising sawdust etc. and for treating gases and vapours with finely-divided substances. (162,769.) May 19.  
 3860 (1920). Firth, Blakeley, Sons and Co., and Blakeley. Gas purifiers. (163,095.) May 25.  
 5563 (1920). Glover, West, and West's Gas Improvement Co. Vertical retort setting for the destructive distillation of coal etc. (163,150.) May 25.  
 7180 (1920). Evans. Refining crude oil etc. by distillation. (163,173.) May 25.  
 7181 (1920). Evans. Crude oil refining processes and apparatus. (162,873.) May 19.  
 10,835 (1920). British Thomson-Houston Co. See X.  
 13,718 (1920). Sawtell. Destructive distillation of wood. (143,545.) May 25.  
 16,539 (1920). Lotz. Burning pulverulent fuels. (162,958.) May 19.

## III.—TAR AND TAR PRODUCTS.

### APPLICATIONS.

- Adam, Galbraith, Shannon, and Siderfin. Manufacture of amines from phenolic compounds. 13,664. May 13.  
 Plauson's (Parent Co.), Ltd. (Plauson). 14,188. See XX.

### COMPLETE SPECIFICATIONS ACCEPTED.

- 27,221 (1919). Sasa. Manufacture of phthalic anhydride. (140,051.) May 19.  
 79 (1920). Commmin. Treating pitch. (162,727.) May 19.  
 10,924 (1920). Tindale. Distillation of tars and treatment of residual pitches. (163,199.) May 25.

## IV.—COLOURING MATTERS AND DYES.

### APPLICATIONS.

- Barritt. Azo dyestuffs and intermediates. 13,764. May 17.

- Farbenfabr. vorm. F. Bayer u. Co. Manufacture of copper compounds of substituted azo dyestuffs. 13,624. May 13. (Ger., 14.6.20.)  
 Johnson (Badische Anilin- u. Soda-Fabr.). 14,161. See XIII.

### COMPLETE SPECIFICATION ACCEPTED.

- 23,799 (1919). Pope, and Scottish Dyes, Ltd. Production of colouring matters. (162,687.) May 19.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

### APPLICATIONS.

- Boyer. Treatment of fabrics, paper, etc. 13,509. May 12.  
 Eylich and Schreiber. Removing ink from print paper. 13,850. May 17.  
 Humphrys. Treating flax, straw, etc. 14,175. May 20.  
 Plauson's (Parent Co.), Ltd. (Plauson). Extraction of wood. 13,758. May 17.  
 Plauson's (Parent Co.), Ltd. (Plauson). 14,187. See XVII.  
 Schülke. Production of artificial multiple-filament threads from cellulose solution. 13,592. May 13. (Ger., 17.5.20.)  
 Schülke. Production of wool-like yarns from artificial threads. 14,033. May 19. (Ger., 20.5.20.)

### COMPLETE SPECIFICATIONS ACCEPTED.

- 2120 (1920). Deuts. Celluloid Fabrik. Manufacture of cellulose compounds. (138,116.) May 19.  
 2995 (1920). Bouillon and Worms. Treatment of cellulose filaments, threads, or films. (162,759.) May 19.  
 3951 (1920). Courtaulds, Ltd., and Stokes. Washing or otherwise treating with liquids artificial filaments etc. (163,099.) May 25.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### APPLICATIONS.

- Ashworth. Dyeing-apparatus. 14,002. May 19.  
 Brandwood and Brandwood. Bleaching, dyeing, etc. yarns wound in cheese form. 13,571. May 13.  
 Britton and others. 13,871. See XIV.

### COMPLETE SPECIFICATIONS ACCEPTED.

- 32,733 (1919). Kershaw and Cole. Machines for dyeing, mordanting, bleaching, etc. wool, slubbing, yarn, etc. (162,720.) May 19.  
 3537 (1920). Arent. Fireproofing and/or waterproofing treatment of materials. (138,641.) May 19.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

### APPLICATIONS.

- Crosland. 13,676. See IX.  
 Gaillard. Manufacture of sulphuric acid. 13,210. May 10.  
 Harter and Oehrich. Production of a contact mass for the catalytic synthesis of ammonia. 13,656. May 13.  
 Minami Manshu Tetsudo Kabushiki Kaisha. Manufacture of anhydrous chlorides of alkaline-earth metals. 13,290. May 10. (Japan, 15.5.20.)  
 Nitrogen Corp. 13,161. See I.  
 Nitrogen Corp. Preparation of gas mixtures and ammonia. 13,162. May 9. (U.S., 8.5.20.)  
 Nitrogen Corp. Production of hydrogen and ammonia. 13,812. May 17. (U.S., 14.5.20.)  
 Nitrogen Corp. Production of hydrogen from hydrocarbons. 13,813. May 17. (U.S., 25.5.20.)

Soc. Anon. de Prod. Chimiques Etabl. Malétra. Continuous production of sulphates. 14,035. May 19. (Fr., 25.5.20.)  
Wilton and Wilton. Manufacture of sulphate of ammonia. 13,261. May 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

2838 (1920). Edwards. Manufacture of arsenical compounds. (162,747.) May 19.  
6206 (1920). Ellis (Foundation Oven Corp.). Purification of ammonium salts. (163,162.) May 25.  
13,579 (1920). McKee. Manufacture of hydrogen and zinc oxide. (163,210.) May 25.  
17,453 (1920). Röhm. Production of a solid non-hygroscopic iron salt. (146,216.) May 19.  
18,165 (1920). Collin A.-G. Discharging ammonium sulphate from saturator baths. (145,781.) May 19.

## VIII.—GLASS; CERAMICS.

## APPLICATIONS.

Clark. Moulding and annealing glass. 13,346. May 11. (U.S., 11.5.20.)  
Feldenheimer and Plowman. Treatment of clay. 13,172. May 9.  
Plauson's (Parent Co.), Ltd. (Plauson). Refining china clay etc. 14,189. May 21.  
Wolf. Cementing procelain. 13,413. May 11.

## IX.—BUILDING MATERIALS.

## APPLICATIONS.

Crosland. Kilns for calcining lime, cement, plaster, ores, etc. 13,676. May 13.  
Hildyard. Production of imitation wood. 13,504. May 12.  
Rowse. Manufacture of roofing and building materials. 14,234. May 21.  
Whithy. Imparting a highly-glazed surface to artificial stone. 13,251. May 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

20,130 (1919). Crawford. Manufacture of bricks. (162,683.) May 19.  
34,876 (1920). Alletson. Kilns for burning blue or other bricks. (163,251.) May 25.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

## APPLICATIONS.

Ashcroft and Lacell. Treatment of ores etc. 13,605-6. May 13.  
Ashcroft and Lacell. Production and purification of lead. 13,607. May 13.  
Boover and District Chemical Co. Flux for welding or depositing metals electrically. 13,181. May 9.  
Brunskill. Treatment to obviate rusting or oxidation of iron or steel surfaces. 13,235. May 10.  
Crosland. 13,676. See IX.  
James and Star Electric Accessories, Ltd. Casting metals of high melting-point. 13,783. May 17.  
Jones. Coating metal plates. 13,268. May 10.  
Marino. Removing rust, grease, paint, etc. from iron and steel. 14,164. May 20.  
Minerals Separation, Ltd., and Vautin. Treatment of ores. 13,283. May 10.  
Small. Production of an alloy. 13,158. May 9.  
Soc. d'Electro-Chimie et d'Electro-Métallurgie. Obtaining deposits of iron by electrolysis. 13,621. May 13. (Fr., 9.8.20.)  
Soc. d'Electro-Chimie et d'Electro-Métallurgie. Obtaining metallic electrolytic deposits easily detachable from the cathode. 13,622. May 13. (Fr., 13.6.20.)

## COMPLETE SPECIFICATIONS ACCEPTED.

19,028 (1919). Browning. Extraction of metals from solutions or separation of metals. (162,682.) May 19.  
25,116 (1919). Lane and Williams. See XII.  
32,571 (1919). Stören and Johanson. Treatment of poor iron ores containing weakly magnetic or non-magnetic oxygen compounds of iron. (162,718.) May 19.  
22 (1920). Wright. Smelting or reduction of iron ore. (162,725.) May 19.  
3181 (1920). Eklund. Recovery of tin from stanniferous waste products. (138,622.) May 19.  
3594 (1920). Heraeus Ges. Vacuum melting and refining of metals and alloys. (138,648.) May 19.  
3672 (1920). Lang. Furnace for making iron and steel. (162,808.) May 19.  
3731 (1920). Heraeus Ges. Purifying and refining iron. (138,651.) May 19.  
4064 (1920). McCaffery. Bessemerising iron. (138,900.) May 19.  
4100 (1920). Jackson (Forge Products Corp.). High-speed steel and process of forming and forging the same. (163,100.) May 25.  
10,835 (1920). British Thomson-Houston Co. (General Electric Co.). Tungsten alloys especially for electric lamp filaments. (162,907.) May 19.  
11,934 (1920). Electrolytic Zinc Co. of Australasia. Recovery of zinc by electrolysis. (155,792.) May 25.  
12,432 (1920). Vos. Alloys. (162,917.) May 19.  
13,831 (1920). Krupp A.-G. Manufacture of hard cast-iron articles. (143,554.) May 25.  
15,645 (1920). Heraeus Ges. Treating metal used in the manufacture of parts of vacuum tubes. (144,890.) May 25.  
26,768 (1920). Kilby and Bacon. Recarburising molten steel in an open-hearth furnace. (162,994.) May 19.

## XI.—ELECTRO-CHEMISTRY.

## APPLICATIONS.

Boover and others. 13,181. See X.  
Burgess Battery Co. Dry cell. 13,661-2. May 13. (U.S., 15.5.20.)  
Coley and Frampton. Regeneration of spent dry cells. 14,149. May 20.  
Garbutt. Electric accumulators. 13,398-9. May 11.  
Hancock and Hancock. Electric furnaces. 13,497. May 12.  
Lodge Fume Co., Ltd., Lodge, and Stallard. Electrical precipitation apparatus. 14,230-1. May 21.  
Niblett. Apparatus for producing electrolytic compounds. 13,192. May 10.  
Niblett. Primary and secondary batteries. 13,681. May 17.  
Soc. d'Electro-Chimie et d'Electro-Métallurgie. 13,621-2. See X.

## COMPLETE SPECIFICATIONS ACCEPTED.

11,934 (1920). Electrolytic Zinc Co. See X.  
13,671 1920. Merrmann. Manufacture of material for use as electrical insulator. (153,884.) May 19.

## XII.—FATS; OILS; WAXES.

## APPLICATION.

Pineger. Detergent. 13,114. May 9.

## COMPLETE SPECIFICATIONS ACCEPTED.

25,116 (1919). Lane and Williams. Treatment of waste grease from tin works and other works. (163,056.) May 25.  
25,355 (1919). Williams and Moseley. Soaps, polishes, etc. (162,691.) May 19.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

Anderson and MacLaurin. Preparation of synthetic resins. 13,744. May 17.  
Ivinson and Roberts. Enamel or varnish. 13,912. May 18.

Johnson (Badische Anilin u. Soda-Fabrik). Production of pigment colours. 14,161. May 20.  
Lefebvre. Obtaining an oil having the chemical properties of oil of turpentine. 13,160. May 9.

#### COMPLETE SPECIFICATION ACCEPTED.

13,900 (1920). Jerone. Waterproof paints. (146,986.) May 19.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### APPLICATION.

Britton, and Griffiths Bros. and Co. Preparation of rubberised fabrics and rubber goods. 13,871. May 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

16,560 (1920). Farrell Foundry and Machine Co. Mixing or masticating rubber etc. (147,547.) May 25.

13,915 (1920). Goodyear Tire and Rubber Co. Manufacture of rubber products. (146,992.) May 19.

19,585 (1920). Waitz. Recovering caoutchouc and fabric from rubbered fabric. (147,628.) May 25.

### XV.—LEATHER; BONE; HORN; GLUE.

#### COMPLETE SPECIFICATIONS ACCEPTED.

63 (1920). Melamid. Manufacture of artificial tanning substances. (137,323.) May 19.

4080 (1920). Marks (Chicago Process Co.). Liming and dehairing hides. (163,109.) May 25.

### XVI.—SOILS; FERTILISERS.

#### COMPLETE SPECIFICATION ACCEPTED.

5100 (1920). Silberrad. Artificial manures. (163,137.) May 25.

### XVII.—SUGARS; STARCHES; GUMS.

#### APPLICATION.

Plauson's (Parent Co.), Ltd. (Plauson). Saccharification of wood and like cellulosic materials. 14,187. May 21.

#### COMPLETE SPECIFICATION ACCEPTED.

19,707 (1920). Aumann. Preparation of sugar beets suitable for making marmalades, extracts, wines, beers, etc. (147,713.) May 19.

### XVIII.—FERMENTATION INDUSTRIES.

#### APPLICATION.

Bryant. Aeration of brewers' wort etc. 13,924. May 18.

#### COMPLETE SPECIFICATIONS ACCEPTED.

2057 (1920). Goldschmid and Migeon. Treatment of peat, distillers' wash, etc. (162,738.) May 19.

19,707 (1920). Aumann. See XVII.  
22,102 (1920). Bensley. Portable dry yeast. (162,978.) May 19.

### XIX.—FOODS; WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

Baker, and Fluorescent Materials, Ltd. Charging water etc. with radium emanation. 13,873. May 17.

Pennell. Filtration of turbid water etc. 13,411. May 11.

Prentice and Stevenson. Sterilisation of milk etc. 13,537. May 13.

Weddingen. 14,147. See I.

#### COMPLETE SPECIFICATIONS ACCEPTED.

2972 (1920). Koppers Co. Purification of liquids. (139,159.) May 25.

8229 and 8330 (1920). Wood. Food compositions. (140,462-3.) May 19.

19,707 (1920). Aumann. See XVII.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### APPLICATIONS.

Chem. Fabr. vorm. Sandoz. Manufacture of a new preparation of hexamethylenetetramine. 13,981. May 18. (Switz., 12.6.20.)

Dreyfus. Manufacture of organic products. 13,472. May 12.

Hoffmann-La Roche u. Co. Manufacture of soluble double compounds of 1-allyl-3,7-dimethylxanthine. 13,674. May 13. (Switz., 2.7.20.)

Hoffmann-La Roche u. Co. Manufacture of 1-allyl-3,7-dimethylxanthine. 13,675. May 13. (Switz., 2.7.20.)

Lefebvre. 13,160. See XIII.

Mills. Blood coagulation mediums. 13,276. May 10. (U.S., 6.5.20.)

Plauson's (Parent Co.), Ltd. (Plauson). Manufacture of ichthyol oil and ichthyol preparations etc. 14,188. May 21.

Soc. Anon. de Prod. Chim. Etabl. Malétra. Production of acetaldehyde from acetylene. 14,049. May 19. (Fr., 15.6.20.)

Soc. Chim. des Usines du Rhône. Compressed vanillin. 14,244. May 21. (Fr., 17.2.21.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

16,211 (1920). Badische Anilin- u. Soda-Fabrik. Manufacture of urea. (145,060.) May 25.

31,000 (1920). Penfold. Manufacture of serum from animals. (154,886.) May 25.

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### APPLICATIONS.

Camiller and Hay. Manufacture of multicolour screens, films, etc. for natural colour cinematography etc. 13,637. May 13.

Christensen. Multicolour screens for photography. 13,524. May 12. (Denmark, 15.5.20.)

Lage. Production of coloured photographs. 14,031. May 19.

### XXII.—EXPLOSIVES; MATCHES.

#### COMPLETE SPECIFICATION ACCEPTED.

27,032 (1919). Brock and Butt. Pyrotechnics, matches, percussion caps, fuses, etc. (162,697.) May 19.

### XXIII.—ANALYSIS.

#### APPLICATION.

Dale. Apparatus for analysis and recording of gases. 13,447. May 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

4228 (1920). Fairweather. Gas calorimeters. (162,826.) May 19.

20,902 (1920). Arndt. Gas-analysing apparatus. (148,998.) May 19.

**I.—GENERAL; PLANT; MACHINERY.**

*Protective linings and coverings for chemical plant.*  
F. Schüler. Chem.-Zeit., 1921, 45, 315–316.

Most of the coverings in use for protecting chemical plant present some disadvantages, and some have a very limited application. Metallic coatings applied galvanically have a short life owing to their thinness and porosity. Sheet metal linings cannot be used with vacuum plant, as the air between them and the walls of the vessel tends to loosen them; on the other hand, a homogeneous layer of lead is satisfactory in many cases. Coating vessels with paints or varnishes is not successful when these are subject to mechanical friction from stirrers etc. or to raised temperature. Enamelled apparatus has frequently considerable durability, but its liability to chip is a drawback. Earthenware linings have found an extensive use, especially with acids, and the sections are sometimes applied in double layers, or after previous coating with lead; objections to the use of this material are its thickness, which materially reduces the capacity of vessels, and the fact that it cannot be used for lining tank covers, or for such purposes as in heating coils, where it is liable to fracture. Thin glass plates have been found to be very resistant and in many ways superior to earthenware, inasmuch as they may be used for covering parts of apparatus and also for lining jacketed vessels, for which earthenware is unsuitable owing to its thickness. Glass linings are not liable to fracture by sudden changes of temperature.—W. J. W.

*Humidity control by means of sulphuric acid solutions, with critical compilation of vapour pressure data.* R. E. Wilson. J. Ind. Eng. Chem., 1921, 13, 326–331.

THE use of sulphuric acid solutions of definite composition affords the most convenient means of obtaining atmospheres of known humidity; the air is bubbled through three successive vessels containing sulphuric acid of the necessary strength. The acid in the first vessel usually needs changing when its volume has altered by 3–4%. A graph is given showing the strength and gravity of sulphuric acid solutions which will give any desired humidity to air; for instance, reference to the graph shows that a relative humidity of 50% at 25° C. is obtained with 43.4% sulphuric acid (sp. gr. 1.329). Formulae are also given for calculating the vapour pressures from one temperature to another when it is desired to employ other temperatures than those shown on the graph.—W. P. S.

**PATENTS.**

*Cooling viscous liquids; Process for — and apparatus therefor.* Soc. des Condenseurs Delas. E.P. 142,454, 19.4.20. Conv., 30.4.19.

THE viscous liquid (e.g. heavy oil) flows in the form of threads through suitable nozzles on to a vertical series of water-cooled pipes, the oil spreading over the whole surface of the pipes but re-forming into drops or threads upon the underside and dropping upon the next pipe. In order to ensure that there shall be no mixing of the liquid and the cooling water as a result of leakage, the header plates supporting the tubes at each end may be double, with a space between.—B. M. V.

*Electrical gas purification.* Metallbank n. Metallurgische Ges. A.G., and J. E. Lilienfeld. E.P. 145,477, 21.6.20. Conv., 8.8.18.

THE generation of ions in the electrical discharge is increased by the use of pulsating undamped direct current voltage, the frequency of pulsation

ranging from 250 to 1500 cycles per sec., and the number of cycles being adapted to the capacity of the high tension circuit, so that the pulsations are not smoothed out by the capacity of the circuit. The pulsating direct current voltage is suitably obtained by superimposing upon a constant direct current voltage an oscillation of a considerably greater frequency than that of the usual technical alternating current.—J. S. G. T.

*Disintegrators or pulverisers.* J. Bray. E.P. 148,551, 10.7.20. Appl., 28.2.19.

IN a Carr's disintegrator, to prevent accumulation of partly ground material in the fine grinding chamber, the faces of the outermost ring of percussion members are inclined to throw the material inwards, the faces of the next ring of percussion members being oppositely inclined. In addition to the screens in the grinding track others may be arranged at the side thereof and a current of air used to draw out the finely ground material.

—B. M. V.

*Centrifugal machines.* P. T. Sharples. E.P. 148,753, 3.7.20. Conv., 30.7.19.

THE material is fed to, and solids discharged from, the bowl through an axial hole in a boss at the bottom. The boss and the bowl are steadied by a drag-bearing permitting slight radial movement and contained in a conical plug easily removable from the outer casing of the machine. The plug also contains means for lubrication of the bearing and a nozzle for introducing the material upwards through the bottom.—B. M. V.

*Centrifugal separator.* E. D. Gray, Assr. to Standard Oil Co. of California. U.S.P. 1,375,506, 19.4.21. Appl., 2.9.20.

THE top of the bowl, forming a closed chamber, is provided with a peripheral pocket. An axial inlet pipe enters near the bottom of the chamber, and a substantially axial outlet pipe leads from a point above the pocket. An outlet from the top of the bowl near its outer edge leads to a chamber forming a casing around the bowl.—H. H.

*Acid-gas or similar substance mains; Pipe connections for —.* P. L. Pfannenschmidt. E.P. 149,684, 31.7.20. Conv., 31.7.19.

TO avoid destruction of the jointing-material of horizontal mains by condensed acid, one pipe end extends beyond the jointing material into an enlarged part of the other pipe from which the condensed acid may be removed. Two similar pipe ends may be symmetrically fitted within a connecting piece jointed to each and formed with an enlargement for collecting the condensed acid.

—H. H.

*Filters.* J. Miller, and G. Fletcher and Co., Ltd. E.P. 161,993, 5.5.19.

A FILTER as described in E.P. 158,387 (J., 1921, 203A) is provided with a stop-cock at the bottom to remove sediment while the filter is in operation and with another cock, preferably leading to the outlet trunnion, through which the residual filtered liquor in the filter can be removed before opening the filter to change the filtering medium. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 23,585 of 1895.)—B. M. V.

*Filters; Production of very fine [ultra] —.* E. de Haën, Chem. Fabr. "List," G.m.b.H. G.P. 334,063, 27.1.20.

IN the preparation of ultra-filters by deposition from a colloidal solution upon a supporting layer,

which may contain a coagulating agent, the colloidal solution (colloidion) is treated with a coagulating agent (water) before it is poured on the supporting layer (filter paper). The permeability of the filter will vary according to the amount of water added and according to the vapour pressure of the water; addition of hygroscopic substances such as calcium chloride or glycerin has a favourable influence on the quality of these ultra-filters.—J. H. L.

*Filter pulp; Process and apparatus for washing* — E. Steuer. G.P. 334,113, 17.6.19.

THE pulp is subjected to the action of water which has been charged under pressure with air or gas. The gas in escaping from solution, loosens the fibres and carries them to the surface. The "gas emulsion" may be produced chemically, e.g., by the action of acids on suitable substances below, in, or above the filter pulp. The apparatus comprises a vessel provided with a perforated false bottom and divided ducts below the latter connected with a pressure vessel in which the washing liquid is saturated with air or gas.—J. H. L.

*Stills; Entrainment separator for* — J. A. Philip. E.P. 162,000, 15.9.19 and 29.1.20.

A SEPARATOR for entrained liquid consists of a number of frusto-conical plates placed close together, with their small ends downwards, in the vapour space of a still, so that any entrained liquid will be caught on the surface of the plates and run back into the distilling liquid. The separator is provided with a conical or domed roof so that any liquid which condenses after passing the separator will be led to a gutter and thence outside the still. The device is specially suitable for mineral oil stills.—B. M. V.

*Liquids and gases; Apparatus for bringing into intimate contact*. W. C. Holmes and Co., Ltd., D. M. Henshaw, and J. Whittell. E.P. 162,166, 1.4.20.

IN a scrubber consisting of a number of brush rings secured back to back in pairs to a series of discs rotating between other fixed discs and dipping into the washing liquid, the rotating discs are perforated between the brush rings and the shaft, and passages are formed through the fixed discs alternately within and without the brush rings. The gas current splits into two streams which pass through the brushes of each pair in the same direction and through adjacent pairs in opposite directions.—B. M. V.

*Separating floating and other impurities in suspension from liquids; Apparatus for* — C. G. Petree. E.P. 162,206, 4.6.20.

THE liquid to be treated, e.g., defecated sugar juice, is delivered tangentially upon a gallery surrounding a central circular hole in the cover of a conical settling vessel and flows through the hole into a cylinder extending from the cover beneath the surface of the liquid in the vessel. This cylinder is provided with an opening near its upper end for the escape of liquid, which is thereupon directed by a volute wall along a path of considerable length to a scum deflector and an overflow for the clear liquid. The scum is intercepted by a plate extending above and below the surface of the liquid and is directed by mechanically-actuated blades to an outlet.—H. H.

*Drying finely divided materials*. H. F. Chappell. U.S.P. 1,375,080, 19.4.21. Appl., 14.11.18.

THE material to be dried is fed on to one end of a travelling platform which is enclosed in an outer chamber. Heating gases which pass through a flue under the platform provide the necessary heat for the drying operation.—A. K.

*Dryer*. E. B. Ayres, Assr. to Procter and Schwartz, Inc. U.S.P. 1,376,244, 26.4.21. Appl., 27.5.20.

A casing is divided into a heating and two drying compartments, and a fan situated in one of the latter circulates air through all three compartments.—B. M. V.

*Drying kiln*. S. E. Cornish, Assr. to H. H. Plummer. U.S.P. 1,376,319, 26.4.21. Appl., 20.1.20.

A KILN is provided at one side with fans which direct air through flues to points on the opposite side of the kiln intermediate between the inlets to the fans, so as to create a continuous circuitous movement of air in the kiln.—B. M. V.

*Dryer; Column — consisting of separate cells*. G. Zimmermann. G.P. 333,943, 5.7.17.

IN a column dryer consisting of a number of cells each of which is connected with the one below it by a funnel-shaped constriction, the walls of the cells are provided with oblique openings for the current of air which traverses the cells. The descending material travels only very slowly in the cells themselves, and owing to the wide openings in their walls the transverse air currents have a relatively small velocity and encounter only small resistance.—J. H. L.

*Fire-extinguishing composition*. P. A. Crosby, R. R. Matthews, and R. B. High, Assrs. to Roxana Petroleum Co. U.S.P. 1,375,779, 26.4.21. Appl., 8.8.19.

THE constituents of the composition are sodium bicarbonate, a phosphoprotein, and water.—B. M. V.

*Catalytic material [nickel]; Process of making* — S. Drucker, and W. Sieck, jun. U.S.P. 1,375,368, 19.4.21. Appl., 7.11.19.

NICKEL carbonate is precipitated from a nickel salt solution, washed, mixed with a powdered inert non-reducible material, and the mixture dried without calcining and reduced in an atmosphere of hydrogen.—H. H.

*Alcohol and ether [vapours] from admixture with air; Process for the recovery of* — E. Bind-schedler. U.S.P. 1,376,069, 26.4.21. Appl., 2.8.20.

ALCOHOL is removed from the mixed vapours by intimate contact with water, and ether absorbed from the residue by means of concentrated sulphuric acid.—J. S. G. T.

*Tube evaporator; Rotary* — A.-G. der Chemischen Produkten-Fabrik Pommerensdorf. G.P. 333,304, 22.12.17.

To avoid vibration, and failure of the driving gear, a tube evaporator is placed eccentrically in its fire chamber, and is so arranged that its weight is equally distributed on both sides of the drive.—W. J. W.

*Separation of small quantities of materials from liquids and gases*. H. Bechhold. G.P. 335,303, 31.8.18.

SOLID impurities are removed from a liquid or gas by adsorption on a suitable medium, such as charcoal or fuller's earth, provided with a coating of insoluble material which enters into combination with the impurities to be removed. For example air is freed from chlorine by filtration through charcoal coated with 2% of mercury.—C. I.

*Gas-mixtures; Separation of — by means of an internally heated column*. G. Petzel. G.P. 335,115, 10.4.14.

THE column is fitted with an internal heating coil

The highly compressed gas mixture, e.g., air, before entering the coil, is allowed to expand to the pressure suitable for operating the column, the cold produced by the expansion serving to cover the losses through radiation etc.—C. I.

*Pulveriser mills.* Fuller-Lehigh Co., Assees. of J. W. Fuller. E.P. 145,497, 21.6.20. Conv., 1.6.18. SEE U.S.P. 1,345,082 of 1920; J., 1920, 564 A.

*Separation of gas mixtures; Process for the —.* G. Petzel. E.P. 148,302, 9.7.20. Conv., 9.4.14. SEE G.P. 335,115 of 1914; preceding.

*Separating suspended matter from liquids; Process and apparatus for —.* A. F. Meston. E.P. 162,390, 28.1.20.

SEE U.S.P. 1,334,160 of 1920; J., 1920, 355 A.

*Grinding-mills [; Open-pan —] for grinding flint, china stone, and other materials.* W. F. Malkin. E.P. 162,481, 8.3.20.

*Protecting walls of reaction vessels.* E.P. 140,083. See VII.

*Removing dust from gases.* E.P. 147,020. See X.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Complete gasification; Experiments in —.* F. H. Robinson. N. Eng. Gas Managers' Assoc., 6.5.21. Gas World, 1921, 74, 363—371.

EXPERIMENTS were carried out at Harrogate on a battery of eight retorts, 14 ft. long by 22 in. by 6 in., with a water-gas generator at one end, the whole installation being accommodated within the roof of the retort-house. The ends of the retorts on the charging side had the ascension pipes connected in the usual way, whilst the other ends terminated in a chamber above the generator. Primary and secondary air could be admitted or shut off simultaneously by means of a piston valve which was also connected with the steam jet, the steam supply to the blowing engine, and the stack valve, so that only one movement was necessary to change from "blow" to "run," or *vice versa*. During the run, the water-gas passed from the generator into and through the retorts and away on the ascension pipes, the heat of the hot gas being so utilised. The coke was discharged from the various retorts in turn to the generator. A thin air, sp. gr. 1.12, was obtained, which yielded water 62%, light oils to 170° C. 1.6%, 170°—230° 3.4%, 230°—270° 13.8%, 270°—300° C. 7.2%, and pitch 8.8%. It was entirely free from separated naphthalene even on standing overnight. The coal used was a mixture of Yorkshire and Durham containing 2.87% of moisture, and (on a moisture-free basis) 29.50% of volatile matter, 57.00% of fixed carbon, and 13.50% of ash. The setting was connected with the foul mains in the retort house so that the hot water-gas could be enriched with the lighter hydrocarbons from the tar vapour therein. The gas produced contained, in one case, CO, 8.4%; O, 0.2%; illuminants 1.4%; CO 18.9%; CH<sub>4</sub>, 17.42%; H<sub>2</sub>, 39.26%; and N, 14.42%, and had a calorific value of 396 B.Th.U. per cub. ft. (gross). The average CO content over a long period was 29.3%, whilst the CO<sub>2</sub> content was 5.8% and the calorific value was 384 B.Th.U. per cub. ft. The total efficiency of the process as determined on three occasions was 70.3%, 73.3%, and 82.6% respectively, values which are very close to the figures mentioned in the Clerk-Smithells-Cobb report of 1919.—A. G.

*Producers for the gasification of natural fuels with recovery of the by-products.* H. Kosbmieder. Brennstoff-Chem., 1921, 2, 134—135, 150—152.

THREE separate zones may be distinguished in a producer using natural fuels such as wood and peat, viz., the upper zone in which moisture is driven off, the middle zone in which the fuel is destructively distilled, and the lowest zone where the coke is burned. The capacity and height of the producer for most efficient working depend upon the nature of the fuel, and the rate at which it passes through the producer. The variations in the size of the producer required with wood, peat, lignite, and coal are discussed with a view of showing the most efficient dimensions for each type of fuel. The advantages of carbonisation are also discussed for certain fuels.—W. P.

*Gas purification.* W. A. Dunkley and C. E. Barnes. Gas J., 1921, 154, 27—29, 148—150, 204—205, 266—268, 381—382.

IN a co-operative research carried out by the Illinois State Geological Survey Division, the Engineering Experiment Station of the University of Illinois, and the U.S. Bureau of Mines, sixteen gas plants, comprising nearly all the medium-sized plants of the State, including the suburban plants at Chicago, were inspected. Low-sulphur Eastern gas coals were being used in practically all the coal gas plants inspected. The average H<sub>2</sub>S content of the gas entering the purifiers was 250 grains per 100 cub. ft. Six of the eight straight water-gas plants were using low-sulphur coals for generator fuel. The average H<sub>2</sub>S content in the unpurified gas in these plants was 140 grains per 100 cub. ft. Tar was found in appreciable amounts in the gas entering the purifiers in nearly all the plants inspected, and the spent oxide from all the plants contained some tar (6.9% in that from water-gas plants and 3.6% in that from mixed gas plants). Tar seemed in some cases to be responsible for low sulphur absorption. The spent oxides from mixed gas plants contained, on the average, 37.4% of sulphur, whilst those from straight water-gas plants contained 21.7%. Revivification *in situ* was practised by most water-gas plants, but by few mixed gas plants, and only one plant revived in the off box. Little trouble was reported in this plant, and the operating cost was low. Though several purifying installations are arranged for reversible gas flow, or for rotation of boxes, little effort seems to be made to realise the fullest advantage of such arrangements. In very few cases were records kept of the performance of particular batches of oxide, and there was a lack of systematic testing of the purifiers to determine the performance of a batch of oxide. Total purification costs for 1919 varied from 0.5 c. to 2.25 c. per 1000 cub. ft. of gas purified. The authors recommend the establishment of simple but regular testing routine, together with better purification records, and the analysis of fouled oxides for sulphur and tar. In several cases coals of higher sulphur content could be used if existing equipment were rearranged and made more flexible in operation.—A. G.

*Benzene hydrocarbons; Determination of — in coal gas and coke-oven gas.* A. Krieger. Z. angew. Chem., 1921, 34, 192.

THE paraffin method for the absorption of benzene hydrocarbons still remains the best practical method for their determination in coal gas etc., and the bad results recorded by Berl and others (J., 1921, 239A) are due to incorrect methods of working, particularly the use of an insufficient amount of absorbent paraffin. Further improvements in this method are attained by removing the last traces of water from the gas by means of carbide, and by using a freezing mixture instead

of ice. The new method using activated carbon offers no advantage over the paraffin method either in point of accuracy or time required.—G. F. M.

*Petroleum; Determination of chlorides in* —. R. R. Matthews. *J. Ind. Eng. Chem.*, 1921, 13, 325–326.

FIVE hundred c.c. of the oil is mixed in a cylinder with 125 c.c. of acetone, 1375 c.c. of water is added, and the mixture shaken carefully to avoid the formation of an emulsion. When the water-acetone layer has separated, about 400 c.c. is drawn off, filtered, and the chloride titrated with *N*/20 silver nitrate solution.—W. P. S.

*Paraffin; New developments in the separation of* — from lignite tar and its distillates. F. Seidenschur. *Brennstoff-Chem.*, 1921, 2, 49–51, 73–76, 81–86.

THE method in general use at present consists in distilling the tar with the use of steam or vacuum and chilling the dried distillate oils. The wax that crystallises is removed by filter-pressing and submitted to a process of "sweating." A final treatment with sulphuric acid and decolorising earth may be employed. The process is very expensive in labour and materials. So far the principal objects of the industry have been the production of wax, the expressed oil being used as fuel, but recently this oil has served as a source of lubricating oil, large amounts having been used during the war. One method that has been suggested for separating the wax consists in boiling the distillate oils with alcohol, the wax being preferentially soluble and crystallising from the alcoholic solution on cooling. The separation is, however, very imperfect, the wax containing much oily matter. A number of patents have been taken out for the application of the Edeleanu method, since liquid sulphur dioxide is a fairly good solvent for unsaturated and aromatic compounds, the paraffinoid components remaining undissolved. By the addition of aqueous pyridine or pyridine bases to lignite tar the paraffin is first precipitated, and subsequently other oils, the tar acids remaining dissolved in the aqueous pyridine, and being finally recovered by distillation. Erdmann has found that the addition of two volumes of acetone in conjunction with moderate chilling precipitates the paraffin almost completely. In all these cases the neutral oils, freed from paraffin, may be used as lubricating oils. The author has found that certain mixtures of alcohol and benzol are quite as effective as acetone in precipitating the wax, hard and soft paraffin being capable of preparation in one operation; the mixture is, moreover, sufficiently non-volatile to obviate the losses involved with acetone. A number of experiments at 0° and –20° C. with acetone and with alcohol-benzol show the solubility of paraffin wax in these liquids and the quality of the wax precipitated by them. The apparatus and method of procedure adopted for working up considerable quantities of lignite tars into wax and lubricating oils are described.—A. E. D.

*Vaseline and paraffin; Action of oxidising agents on* —. A. Langer. *Chem.-Zeit.*, 1921, 45, 466.

INVESTIGATIONS in regard to the conversion of hydrocarbons into fatty acids were made with vaseline oil, vaseline, and liquid and solid paraffin, using oxidising agents such as potassium permanganate in acid and alkaline solution, sodium hypochlorite and permanganate, manganese dioxide and hydrochloric acid, and nitric acid. When a mixture of yellow vaseline oil and concentrated sodium hydroxide solution was treated for a few days with permanganate, a supernatant oil was obtained and a residue which was dissolved in dilute sulphuric acid. The oil from this residue was only slightly soluble in sodium carbonate or hydroxide solution;

when heated with concentrated sulphuric acid, it gave a black crystalline mass and evolved sulphur dioxide. It was insoluble in most solvents. Oxidation with permanganate and sodium hypochlorite solution gave no better results. Liquid paraffin behaved in a similar manner to vaseline oil, when treated with alkaline permanganate. When oxidised with acid permanganate solution, undissolved manganese hydroxide remained; the residue after filtration was dissolved in hydrochloric acid and yielded a soft resin. The oxidation product from solid paraffin is of the nature of a salve; with litharge it forms a lead salt which is soluble in ether. From vaseline a hard and a soft resin are produced, the latter containing no substances of an acid character; the yield of saponifiable matter is smaller than with vaseline oil, nor does the product form soluble lead salts as is the case with the product from solid paraffin.—W. J. W.

#### PATENTS.

*Fuel and method of producing same.* L. W. Bates. U.S.P. 1,375,811, 26.4.21. Appl., 22.1.20.

ASPHALT and free-carbon particles in a liquid hydrocarbon are stabilised by mixing "coal distillate or an equivalent peptising substance" with the hydrocarbon and "blending the components and simultaneously peptising the particles with heat treatment between normal temperature and 95° C." —L. A. C.

*Peat and similar material; Manufacture of briquettes from* —. S. Henlein and A. Hopf. G.P. 332,890, 27.11.19.

THE material is first passed through an extrusion press to ensure mixing of its various layers, and is then subjected to a high pressure to remove mechanically combined water, after which it is ground and passed through a rolling mill at a temperature of 200°C., by which means the moisture is reduced to a small percentage. The pasty material obtained may be again pulverised or briquetted without further treatment.—W. J. W.

*Pulverulent fuels; Method of and means for burning* —. R. S. Walker. E.P. 139,158, 28.1.20. Conv., 15.2.19.

THE powdered fuel is fed into the combustion chamber in a thin vertical stream of curtain-like formation, and its combustion occurs as a reverting flame from which the ignition of the entering fuel is maintained. The supply of air for combustion is directed into the chamber from below, so that ash, in falling from the combustion zone, is cooled and slagging is thus avoided. The feed device for the fuel consists of a nozzle with a horizontal slot extending transversely across the chamber.—A. G.

*Coking plants.* Gewerkschaft Emscher-Lippe, and H. Heyn. E.P. 153,313, 2.11.20. Conv., 3.11.19.

THE glowing coke is cooled by the circulation of inert gases through the mass, the gases imparting their heat to a heat-interchange system, e.g., a steam boiler and feed water heater. For example, the coke is loaded into travelling cars which are connected with a blowing system by means of which flue gas is blown through the hot coke, and the hot gases are then passed through two feed water heaters and a steam boiler, travelling on a car which is capable of running along the discharge side of the coking plant.—A. G.

*Coke ovens.* J. Marr, and The Coke Oven Construction Co., Ltd. E.P. (A) 162,045 and (S) 162,236, 16.1.20.

(A) In a coke oven of the type having two parallel horizontal heating flues side by side in each dividing wall between the retorts, an inlet conduit for hot air is provided at the front end of one such flue. A



gas inlet is connected with the conduit, and horizontal ports lead from the other end of that flue to the other flue in the same dividing wall, a second gas supply being connected with the ports, whilst a conduit at the front end of the second flue leads to a discharge conduit for the products of combustion. The flues have longitudinal horizontal partitions extending from the conduit at the front right to the back of each flue. Ports at the rear ends of the flues afford communication from each of the spaces between the partitions in one flue to the spaces at the same level in the return flue. Each space has a gas inlet at the rear end of the flue. Either of the two conduits at the front ends of the two flues acts as the inlet, as desired, and both the conduits are provided with gas inlets. The conduits at the front ends of the two flues, one on each side of each retort, are connected with a single sole channel running longitudinally below the retort for its whole length, with or without a preliminary gas admission at the rear end of the sole channel. (s) The retorts have horizontal heating flues in the walls and regenerators below the retorts, each communicating with the adjacent ends of two flues, the next adjacent regenerator on each side communicating with the other end of one of the above-mentioned two flues and also with the adjacent end of the next flue. Alternate regenerators are employed for the supply of the air and for the exhaust of the waste gases, two transverse main flues communicating one with one set of alternate regenerator chambers and the other with the other set. Means are provided for establishing communication between either main flue and either the chimney or the atmosphere, so that the flow of the heated gases through the flues may be reversed. Recuperator passages are provided in which partially cooled burnt gases issuing from the regenerators are utilised for preheating the combustible gas by conduction of heat through the walls. One or more additional compound flues are provided with passages for the gas to be heated and for the hot burnt products, conduits affording communication between the passages for burnt products at one end of the said compound flue and the chimney and between the other end of the compound flue and the main transverse flues. In the compound flue, passages for gases extend longitudinally through the flue and are built up of blocks, each containing one or more vertical ports which extend transversely to the direction of the passages and baffles to direct the gases passing through the ports to and fro across the longitudinal passages.—A. G.

*Gas-producer.* \* G. E. and G. M. Williams. E.P. 162,455, 21.2.20.

Air and steam are introduced as a circular blow through an annular hollow chamber, surmounted and overhung by an annular water-cooled casing. This directs the blast into the centre of the producer, and the cooling prevents clinker adhering.—C. I.

*Solid and gaseous fuel; Process and plant for the continuous production of — with by-product recovery.* H. Nielsen and J. R. Garrow. E.P. 162,459, 23.2.20.

ONE of a battery of gas producers is supplied with just sufficient fuel to produce such a quantity of gas that the sensible heat of the gas is sufficient to carbonise the charge in a retort at a low temperature. The mixture of producer gas and gas from the retort is treated to separate the low-temperature volatile oils, and is then mixed with the remaining producer gas from the other producers of the battery and treated for the recovery of ammonia as sulphate. The retort is of the revolving type, and is of such construction as to provide sufficient solid fuel for use in all the producers, a measured

quantity being fed to the producer which generates the gas employed for heating the material in the retort.—A. G.

*Hydrocarbon vapours or gases; Process for generating and utilising —.* J. P. Kendall, Assr. to Kendalite Co., Inc. U.S.P. 1,376,100, 26.4.21. Appl., 14.3.18.

To prevent accumulation of carbon deposits in a hydrocarbon vapour generator for intermittently operated burners, barium oxide is heated in the generator in the presence of air until partially converted into barium peroxide. A hydrocarbon liquid containing an oxidising agent is then charged into the generator and the heating is continued to vaporise the liquid and convert a further portion of the barium oxide into peroxide. The vapour is burnt outside the generator to continue vaporisation and to convert the barium peroxide into oxide with simultaneous oxidation of any carbon deposits. As the apparatus cools, a portion of the barium oxide is reconverted to peroxide.—L. A. C.

*Gas producers with inner distillation chamber; Packing for —.* F. Siemens. G.P. 330,278, 30.6.17.

In a gas producer provided with separate outlets for the products of distillation and gasification and with a rotating distillation chamber, loss of gas on the one hand or admixture of air with the gas on the other hand, in the event of defective packing, is prevented by enclosing the joint between the rotating and fixed parts of the apparatus in a chamber filled with the gases generated in the lower part of the apparatus. The gases from the upper part of the apparatus pass out through an annular space between the charging device and the rotating distillation chamber.—L. A. C.

*Gas-producer.* R. Bergmans. G.P. 330,728, 1.1.19.

THE space below the hearth of a gas producer is divided into two compartments by means of a partition. Into one of these, below the combustion chamber, the air for combustion is introduced, whilst the gases from the combustion chamber are passed into the second compartment, which is beneath the gas exhaust chamber. Admixture of oxygen with the combustion gases is thus prevented.—W. J. W.

*Gas and coke; Production of — and recovery of by-products.* W. Zimmermann. G.P. 334,002, 4.7.18.

COAL is heated by passing gas through it until low-temperature tar is formed, after which degasification is continued by means of external heating and a solid coke is obtained.—W. J. W.

*Ammonia; Extraction of — from gases.* E. L. Pease. E.P. 162,314, 29.10.19.

GAS is passed through a pipe along which a dry material containing aluminium sulphate is conveyed in the reverse direction. The absorbent material is prepared by heating shale or clay with sulphur and coke in a gas producer and collecting the ash.—C. I.

*Ammonia and tar recovery process.* J. Van Ackeren, Assr. to The Koppers Co. U.S.P. (A) 1,375,475, (B) 1,375,476, (C) 1,375,477, and (D) 1,375,478, 19.4.21. Appl., (A and B) 21.8.19, (C and D) 25.8.19.

(A) PRODUCER gas is freed from dust, washed while still hot with water, further cooled to remove tar, and washed with acid. (A) Steam is generated by the heat of the gas leaving the producer and used for blowing the producer after passing through a gas-fired superheater. (C) Gas at its saturation temperature is washed with acid and the dilute

mother liquor concentrated with superheated air, which air, thus saturated with moisture, is used in the producer. (b) Hot gas free from dust is blown through condensed ammonia liquor to distil the ammonia, and the mixed still vapours and dust-free gas are freed from tar and moisture in an electrical precipitator and then washed with acid.

—C. I.

*Ammonia and tar recovery process.* J. Becker, Assr. to The Koppers Co. U.S.P. (a) 1,375,483, (b) 1,375,484, and (c) 1,375,485, 19.4.21. Appl., 21, 22, and 25.8.19.

(A) PRODUCER gas is cooled and freed from dust and tar, and the ammonia removed in an acid washer so operated that the gas effects the concentration of the ammonium sulphate liquor. (B) Gas is cooled and freed from tar by a counter current of air and the heated air used to concentrate ammonium sulphate liquor. (C) Superheated air is used to concentrate the sulphate liquor and the air after reheating supplied to the producer.—C. I.

*Sulphur; Removal of — from gases.* N. E. Rambush. E.P. 162,554, 21.5.20.

IN the removal of sulphur from a gas by washing with a suspension or solution of a metallic compound and revivifying the sludge produced with a current of air, a sensible loss of sulphur occurs in the latter stage. To reduce this loss to a minimum the air is circulated, with admission of enough fresh air to maintain the oxygen content at about 8%; or air may be drawn in slowly as the liquid flows down a tower, by chimney draught and by the injector action of the inflowing liquid.—C. I.

*Oil; Recovery of — from substances such as bitumen and shale in situ.* D. Diver. E.P. 162,337, 23.12.19.

Two bore holes close together in the shale or the like contain metal cylindrical casings, one of which is closed and contains a series of electric heating coils, while the other is perforated for a portion of its length at the lower end, and is provided at the top with a cup and an outlet pipe for conveying vapours to a condenser and gas storage plant. The heat generated in the first casing heats the surrounding rock and decreases the viscosity of the oil therein, which flows through the perforations into the second casing, whence it is removed by pumping or other means, while the lighter fractions are volatilised and pass out through the pipe at the top.—L. A. C.

*Liquid fuel; Process of treating —.* E. E. Wickersham. U.S.P. 1,376,180, 26.4.21. Appl., 26.9.20.

By passing electric sparks through liquid fuel, the final boiling point is reduced.—D. F. T.

*Oil mixtures [e.g., shale oil]; Process for separating —.* K. Kubierschky. G.P. 333,294, 2.12.17.

OIL mixtures are dissolved in alcohol of a high strength and the solution is then washed in a counter-stream of water or aqueous alcohol. To separate shale oil (sp. gr. 0.900) into two fractions of sp. gr. 0.93 and 0.84 respectively, the crude oil is washed with an equal quantity of 90% alcohol, the resulting solution having a sp. gr. of 0.866. By introducing water into the upper end of the washing column, a mixture of 80% alcohol and the extract gradually forms at the bottom and can be drawn off, whilst the oil (sp. gr. 0.84) which separates from the extract passes out at the top.

—W. J. W.

*Lubricants; Production of —.* H. W. Klever. G.P. 331,504, 2.2.15.

SMALL amounts of fatty acid salts of alkaline-earth or heavy metals dissolved in coal-tar oils increase their viscosity appreciably and their power of adhesion to metallic surfaces.—W. J. W.

*Lubricants; Manufacture of —.* P. Friesenhahn. G.P. 332,909, 25.7.19.

HYDROGENISED phenols or their derivatives, such as cyclohexanone and its homologues, or cyclohexanol and its esters, may be used, either alone or mixed with other lubricants, as lubricating agents. Cyclohexanol has sp. gr. 0.945, b.p. 160° C., and viscosity 3.5° (Engler), and readily mixes with fats, oils, and hydrocarbons.—W. J. W.

*Crude oil refining processes and apparatus.* J. G. P. Evans. E.P. 162,873, 9.3.20.

SEE U.S.P., 1,366,643 of 1921; J., 1921, 172 A.

*Furnaces for pulverulent fuel [for locomotives].* Motala Verkstads Nya Aktiebolag, and K. H. W. von Porat. E.P. 162,896, 29.3.20.

*Gas producers; Charging devices for —.* K. Linck. E.P. 143,239, 9.7.20. Conv., 2.2.18.

[Gas] retort settings; [Division walls for] vertical —. Drakes, Ltd., and J. W. Drake. E.P. 162,422, 9.2.20.

*Gas producers, furnaces, and the like; Gas regulating and reversing valves for —.* W. Simons and I. B. Evans. E.P. 162,564, 2.6.20.

See also pages (A) 422, *Stills* (E.P. 162,000). 427, *Products soluble in or forming emulsions with water* (E.P. 134,223); *Asphalt substitute* (G.P. 332,888). 435, *Separating slag from coke, etc.* (E.P. 152,642). 449, *Petroleum preparation* (G.P. 334,916).

## IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Adsorbing power of charcoal; Determination of —.* I. M. Kolthoff. Pharm. Weekblad, 1921, 58, 630—656.

FREUNDLICH's rule, that the order in which materials are adsorbed is almost independent of the nature of the solid phase, is found to hold for five kinds of charcoal tested with many different substances. The order of the adsorbing capacity is:—Blood charcoal (M)>vegetable carbon (M)=medicinal carbon (M)>bactanate >norite (M indicates Merck's preparation). A test of adsorptive power is the weight of mercuric cyanide or phenol adsorbed by 1 g. of charcoal from 100 cc. of N/10 solution. (Cf. J.C.S., June.)—S. I. L.

### PATENTS.

*Decolorising carbon; Method of making —.* C. S. Hudson. E.P. 139,156, 20.1.20. Conv., 15.2.19.

CARBONISABLE organic material is mixed with a fusible acid, e.g., phosphoric acid, in the proportion of not less than 0.5 pt. (e.g., 2 pts.) of acid to 1 pt. of organic material, and the mixture is carbonised at 375° C. or over.—A. G.

*Decolorising charcoal; Process for the preparation of —.* De Bruyn, Ltd., and C. Revis. E.P. 162,117, 6.2.20.

FILTER-PRESS residues from the refining of edible oils, such as cottonseed oil and the like, are carbonised to produce a dry, porous carbonaceous material. The carbonisation takes place with the admission of a regulated quantity of air and the carbonised product is washed with a mineral acid

in order to neutralise the alkaline salts. After neutralisation, the mixture is formed into cakes and again heated to expel moisture and excess acid.—A. G.

*Distilling carbonaceous material; Apparatus for* — W. P. Perry. E.P. 162,136, 25.2.20.

An apparatus for distilling carbonaceous material consists of one structure divided by vertical or slightly inclined partitions into three chambers, the middle one, the distilling chamber, having a number of openings into the gas distributing chamber and the collecting or condensing chamber on either side. The two latter chambers have each a capacity equal to or greater than that of the distilling chamber.—A. G.

*Carbonising apparatus.* H. N. McLeod. U.S.P. 1,375,714, 26.4.21. Appl., 13.12.20.

A NUMBER of chambers are so arranged that the discharge end of one chamber leads into the receiving end of the next chamber in the series. The chambers are divided into two series with separate heating circuits, the heating circuit of the second set of chambers comprising a primary source of heated gas and conduits receiving the heated gas, the walls of the conduits being in contact with the material in the chambers of the second set. The flow of the heated gas through the conduits is in counter-current to the passage of the material through the chambers of the second set. Condensing apparatus is connected with the last chamber of the second set. Conduits having walls in contact with the material in the chambers of the first set receive the heated cooling medium from the jackets of the condenser and convey it in counter-current to the material to be carbonised through the chambers of the first set. The material to be carbonised is conveyed mechanically through the chambers of the first set and then through the chambers of the second set.—A. G.

*Distillation; Furnace system for dry* — T. M. U. von Post. U.S.P. 1,375,943, 26.4.21. Appl., 16.10.19.

The retort proper is inserted in a closed chamber and is provided with a conduit communicating with an inlet at the bottom of the chamber, gases being conducted from the inlet into the top of the retort. The retort is also provided at the bottom with an outlet communicating with an outlet pipe opening into the bottom of the chamber, where means are provided by which the outlet and the inlet are separated from each other by a gas-tight joint. A condenser communicates with the inlet and the outlet.—A. G.

*Distillation of wood, charcoal, or peat; Apparatus and process for continuous* — Köln-Rottweil, A.-G. G.P. 326,227, 21.10.19.

In a process in which the material is distilled either at normal or elevated pressure or *in vacuo*, in horizontal retorts into which it is introduced in counter-current to the heating gas, the charge is first compressed in a suitable machine and then delivered to a screw-conveyor, the latter having a scraper which causes uniform amounts of the pressed material to be introduced into the retort. To ensure rapid and regular heating, the material is kept in motion in the retort; the products of distillation must be rapidly removed.—W. J. W.

*Retort for distillation of carbonaceous materials.* H. Graf. G.P. 328,821, 3.5.19.

The retort, in which the charge is carbonised from the top downwards, is surrounded by a cooling chamber which communicates with it by means of

openings through which the products of distillation pass. A water pipe through the outer chamber serves to condense some of the products, which are drawn off through a pipe at the bottom, whilst the uncondensed gases escape through a pipe which passes centrally upwards through the retort. A high yield of condensed distillation products is thus obtained.—W. J. W.

### III.—TAR AND TAR PRODUCTS.

*Polycyclic phenols from sodium phenoxide fusions.* F. Hofmann and M. Heyn. Brennstoff-Chem., 1921, 2, 147—150.

Sodium phenoxide was heated in a stream of hydrogen or nitrogen at 485°—490° C. Four of the six possible isomeric dihydroxydiphenyls were isolated from the residue. (Cf. J.C.S., July.) —W. P.

*Diphenylamine; Melting point of* — H. Rogers, W. C. Holmes, and W. L. Lindsay. J. Ind. Eng. Chem., 1921, 13, 314—316.

The m.p. of pure diphenylamine was found to be 53.0° C., which is 1° lower than the value given in the literature.—W. P. S.

*Benzene hydrocarbons in gas.* Krieger. See IIa.

*Paraffin from lignite tar.* Seidenschaur. See IIa.

*Aniline bisulphate.* Sabalitschka. See VII.

#### PATENTS.

*Products [from tar] soluble in or forming emulsions with water; Process for the manufacture of* — M. Melamid and L. Grötzing. E.P. 134,223, 22.10.19. Conv., 31.10.17.

THE solid or semi-solid resin-like substances, prepared from soft pitch, coal tar, lignite tar, or the like by extraction with sodium hydroxide solution with subsequent acidification of the extract, are distilled *in vacuo*; the portion distilling above 270° C. (20 mm.) is dissolved in cold sodium hydroxide solution, with or without previous sulphonation, and tar oil or mineral oil is added to the solution. The products are suitable for use as lubricants and as substitutes for soap and Turkey-red oil.—L. A. C.

*Asphalt substitute; Manufacture of an* — Verkaufsvereinigung f. Teerzeugnisse G.m.b.H. G.P. 332,583, 12.8.19. Addn. to 330,970 (J., 1921, 339 A).

ANTHRACENE residues when treated with sulphur yield a product of high tenacity which is suitable for lacquers, briquettes, or road-making.—W. J. W.

*Tar or tar residues, and filling material; Production of a homogeneous mass from* — N. Reif. G.P. 332,941, 3.1.20.

TAR fog is caused to circulate and impinge on the periphery of a moving annular cloud of filling material so as to envelop and penetrate the latter. In order to obtain a product possessing hardness, elasticity, and resistance to heat, the tar, or a mixture of tar and oil, is first oxidised or treated with a halogen or sulphur and then incorporated with the filling material at a suitable temperature.—W. J. W.

*Salts of sulpho-acids; Process for obtaining* — from lignite tar oils. Sudfeldt und Co. E.P. 148,763, 10.7.20. Conv., 14.3.19.

LIGNITE tar oils are treated with sufficient concentrated sulphuric acid to polymerise the acid resins,

i.e., 3 to 5% of their weight, and, after separation of the polymerisation products, with 10 to 20% of concentrated sulphuric acid to sulphonate the oil. After separation of the product into two layers, the oil layer is removed and agitated with an excess of  $N/2$  sodium hydroxide solution to dissolve the sulphonic acids, which are then precipitated from solution by the addition of sodium chloride. The sulphuric acid layer is treated with a little water to remove excess sulphuric acid, the sulphonic acids are dissolved in water, and are precipitated from the filtered solution by the addition of sodium chloride. Sufficient saturated salt solution is first added to precipitate a small fraction of the product together with impurities, and, after separation of the precipitate, the remainder of the product is precipitated by the addition of more salt. The product is purified by treatment with oxidising agents, e.g., permanganates, in aqueous solution.

—L. A. C.

*Naphthylamine sulphonic acids; Manufacture of* —. South Metropolitan Gas Co., and H. Stanier. E.P. 161,859, 25.5.20.

THE separation of the 1.4.8- and 1.3.8-naphthylaminedisulphonic acids from the crude mixture obtained by the sulphonation of naphthylamine is effected by taking advantage of the differences in solubility of the barium salts of the acids. A suitably concentrated solution of the disodium salts is boiled with a quantity of barium chloride sufficient to convert all the acids into their barium salts. Barium 1.4.8-naphthylaminedisulphonate is precipitated, and the liquor is filtered hot. The filtrate and washings are acidified with hydrochloric acid, whereby the acid barium salt of the 1.3.8-disulphonic acid is precipitated, and filtered off hot to secure retention in solution of the corresponding salts of the 2.4.7- and 2.4.8-acids which may be present. Alternatively the barium salt may be added in proportion sufficient to convert only the 1.4.8- and 1.3.8-acids into barium salts, in which case the acid barium salts of the latter acid can be filtered off cold.—G. F. M.

*Sulphur compounds of the thiophene series; Manufacture of* —. H. Scheibler. G.P. 331,793, 17.4.15. Addn. to 327,050 (J., 1921, 173 A).

THE tar oil, after purification with soda-lime, is treated with sodamide or with ammonia and sodium below  $120^{\circ}\text{C}$ .—L. A. C.

*Thionaphthene; Process of separating* — from coal-tar [naphthalene]. Ges. für Teerverwertung m.b.H. G.P. 333,156, 26.2.20. Addn. to 325,712 (J., 1920, 777 A).

THE mixture of sulphonic acids, obtained as described in the chief patent, is distilled with steam at such a temperature that only a small proportion of the naphthalenesulphonic acid is hydrolysed. The distillate, m.p.  $50^{\circ}$ – $60^{\circ}\text{C}$ ., containing 20 to 30% of thionaphthene, is treated with alkali for the removal of phenols, and is then purified by fractional distillation; the fraction  $220^{\circ}$ – $225^{\circ}\text{C}$ . gives a dark-red coloration on treatment in carbon bisulphide solution with sulphuric acid (sp. gr. 1.84). Steam distillation of the solution, after separation of the carbon bisulphide and dilution with water, yields pure thionaphthene.—L. A. C.

*Anthranol; Manufacture of* —. A. G. Perkin, Assr. to British Dyestuffs Corp. U.S.P. 1,375,972, 28.4.21. Appl., 9.6.20.

See E.P. 151,707 of 1919; J., 1920, 743 A.

*Lubricants.* G.P. 331,504, and 332,909. See IIa.

#### IV.—COLOURING MATTERS AND DYES.

*Nitroamines and their derivatives; Some properties of* —. F. M. Rowe. J. Soc. Dyers and Col., 1921, 37, 145–150.

A REVIEW of the author's published work on nitroamines (J., 1913, 417; 1917, 958; 1918, 119 A). The nitrogen analogues of Stilbene Yellow and Mikado Orange can be prepared from *p*-nitroaniline by similar condensations to those which occur in the stilbene series. In the latter case the reaction is facilitated by the presence of negative groups in the *o*-position, but in the former case, sulphonic or nitro groups in the *o*-position with respect to the basic nitrogen exert an impeding effect in condensation, probably owing to a tendency to ring formation. The oxidation of an alcoholic solution of *o*-nitroaniline with neutral sodium hypochlorite results in the formation of 2.2'-dinitroazobenzene, but in presence of alkali benziso-oxadiazole oxide is formed in quantitative yield. The latter compound yields a dinitro-derivative which forms true salts and affords an example of hydrogen in the benzene ring possessed of acidic properties. Dinitrobenziso-oxadiazole oxide, treated with sodium sulphide and sulphur by the condenser method, yields a fine brown sulphide dye of good properties, and thus suggests an outlet for *o*-nitroaniline.

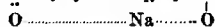
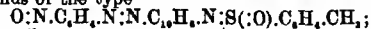
—F. M. R.

*1.8-Dihydroxynaphthalene.* G. Hettler and H. Kretschmann. Ber., 1921, 54, 1098–1107.

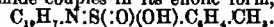
1.8-DIHYDROXYNAPHTHALENE, m.p.  $140^{\circ}\text{C}$ ., is most conveniently prepared by heating 1.8-dihydroxynaphthalene-4-sulphonic acid with sulphuric acid and water at  $150^{\circ}\text{C}$ . It couples with an equimolecular quantity of benzenediazonium chloride in hydrochloric acid solution to 4-benzeneazo-1.8-dihydroxynaphthalene, which does not melt below  $260^{\circ}\text{C}$ .; the same substance is formed in acetic acid or alkaline solution but, in addition, about 10% and 7.5% respectively of products insoluble in alkali are produced. The use of an increased proportion of diazo-compound leads, in acetic acid but not in mineral acid solution, to the production of 4.5-dibenzeneazo-1.8-dihydroxynaphthalene, m.p. about  $238^{\circ}\text{C}$ .; in alkaline solution, an isomeric dibenzeneazo dye appears also to be formed. 4-*p*-Sulphobenzeneazo-1.8-dihydroxynaphthalene dyes unmordanted wool a dark cherry red, a paler shade being produced on aluminium-mordanted wool and dark brown shades on the chrome-mordanted fibre. (Cf. J.C.S. July.)—H. W.

*Azo components; Aromatic acylamines as* —. W. König and K. Köhler. Ber., 1921, 54, 981–987.

DIAZOTISED *p*-nitraniline couples with *p*-toluenesulphon- $\alpha$ - and - $\beta$ -naphthylamides, giving compounds of the type



since reaction does not occur under similar conditions with the corresponding methylnaphthylamides, it appears probable that the naphthylsulphonamide couples in its enolic form,



Carboxyamides can also couple with diazotised *p*-nitraniline but reaction occurs extremely slowly. The auxochromic power of the different groups under consideration diminishes in the order, —OH, —NH.SO<sub>2</sub>.C<sub>6</sub>H<sub>4</sub>CH<sub>3</sub>, —NH.CO.C<sub>6</sub>H<sub>4</sub>, —NH.CO.CH<sub>3</sub>. (Cf. J.C.S., July.)—H. W.

*Isatins; Synthesis of* —. Action of chloraloxime on aromatic amines. T. Martinet and P. Coisset. Comptes rend., 1921, 172, 1234–1236.

In acid solution chloraloxime condenses with aniline to give isonitrosoacetanilide, which when warmed

with sulphuric acid yields isatin. If the aniline is replaced by *p*-chloro-*o*-anisidine hydrochloride the products are in turn 2-methoxy-5-chloroisnitrosoacetanilide, m.p. 190° C., and 4-chloro-7-methoxyisatin. The latter gives the corresponding copper and barium isatates and when condensed with indoxyl yields 4-chloro-7-methoxyindirubin, which with hydrosulphite gives an unstable yellowish-green vat changing almost completely into ordinary indigo.—W. G.

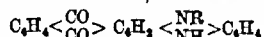
*Isocurcumin. III.* G. Heller. Ber., 1921, 54, 1118—1119.

EXAMINATION of the action of acetylacetone on *p*-hydroxybenzaldehyde and thymolaldehyde in the presence of alcoholic hydrogen chloride has shown that the poor yield of  $\beta$ -isocurcumin obtained from vanillin under like conditions (J., 1914, 544) is due to the decomposition of acetylacetone by the alcoholic acid into acetone with which the aldehyde preferentially condenses.—H. W.

#### PATENTS.

*Anthraquinone series; Manufacture of nitrogenous condensation products of the —.* Badische Anilin- u. Soda-Fabrik. G.P. 329,247, 16.6.14.

1-HALOGENANTHRAQUINONES or derivatives containing a reactive substituent in *o*-position to the halogen atom are heated with *o*-aminodiarilamines or their nuclear substitution products, alone or in an inert solvent or suspension medium, and with or without addition of catalysts and substances capable of combining with acids. Anthraquinonylbenzohydroazines of the formula,



similar to the products described in G.P. 329,246 (E.P. 3347 and 100,581 of 1915; J., 1916, 831), are formed. From 1-chloroanthraquinone, *o*-aminodiphenylamine hydrochloride, potassium acetate, and naphthalene, a product of the composition,  $\text{C}_{24}\text{H}_{14}\text{O}_2\text{N}_2$ , insoluble in dilute hydrochloric acid, and crystallising from acetone in needles with a coppery lustre, is obtained. The same substance is formed from 1,2-dichloroanthraquinone, m.p. 202°—204° C., and from 1-bromo-2-methoxyanthraquinone, whereas from 1,5- or 1,8-dichloroanthraquinone two isomeric monochloro-derivatives of the substance are formed. With 1,4-dichloroanthraquinone both chlorine atoms are replaced, and a blue condensation product, almost insoluble in acetone, is obtained.

*Anthracene derivatives containing nitrogen; Preparation of —.* Preparation of nitrogenous condensation products of the anthraquinone series. Badische Anilin- und Soda-Fabrik. G.P. (A) 330,572, 14.8.14, and (B) 332,013, 16.6.14. Addn. to 329,247 (cf. supra).

(A) ALIZARIN or a heteronuclear hydroxy-substitution derivative (with the exception of 1,2,5,8-tetrahydroxyanthraquinone) is heated with primary aromatic amines or their substitution products with or without addition of stannous chloride. On heating alizarin with aniline and stannous chloride to 170°—180° C., phenylaminococeramidonine is formed, which, after freeing the melt from excess of aniline by hydrochloric acid, is extracted with acetone, from which it crystallises in glistening needles, m.p. 203°—205° C. (uncorr.), soluble in alcohol, nitrobenzene, and aniline with a red, and in concentrated sulphuric acid with a greenish blue colour. Other arylaminococeramidonines from *p*-toluidine and alizarin (m.p. 198°—199° C.), 1,2,6-trihydroxyanthraquinone and *p*-toluidine (m.p. 284°—287° C.), 1,2,7-trihydroxyanthraquinone and *p*-toluidine, 1,2,5-trihydroxyanthraquinone and *p*-toluidine, and 1,2,5,6-tetrahydroxyanthraquinone

and *p*-toluidine, are described. All these products can be used as dyestuffs or as raw materials for the preparation of dyestuffs. Their sulphonic acids dye unmordanted wool in red, violet, and blue shades. (A) Arylaminoanthraquinones, or their nuclear substitution products, containing a reactive substitution group in the ortho-position to the imino-group, are condensed with primary aromatic amines. Thus 1-phenylamino-2-methoxyanthraquinone is produced from 1-bromo-2-methoxyanthraquinone and aniline; it dissolves in sulphuric acid with a yellowish-red and in alcohol with a carmine-red colour. It gives a condensation product with aniline and caustic potash identical with that described in the principal patent.—A. R. P.

*Diazo-azo-hydroxy compounds; Preparation of —.* O. Müller. G.P. 330,832, 7.4.16.

SUFFICIENT alkali carbonate is added to render alkaline a solution containing equivalent quantities of (A) a diazohydroxynaphthalene, or derivatives of the same, such as 1-diazo-5 (or 7)-hydroxynaphthalene, or 2-diazo-8-hydroxy-6 (or 3,6-di)-sulphonic acid, and (B) a highly reactive mono-diazo compound containing a negatively substituted benzene nucleus, such as *p*-nitrodiazobenzene. Alternatively, a solution of (B) is added to a solution of (A) containing sufficient alkali carbonate to render the solution alkaline at the end of the reaction, which is marked by the disappearance of both diazo compounds from the solution. The products couple, e.g., with *m*-toluylenediamine, 1-aminonaphthalene-6 (7)-sulphonic acid, or 1-amino-8-hydroxynaphthalene-4-sulphonic acid, yielding dark blue to black substantive cotton dyes. Diazo-1-hydroxynaphthalene compounds which are capable of reacting with a diazo compound in both alkaline and acid solution, produce in the two cases different diazo-azo compounds, which yield different dyestuffs on coupling.—L. A. C.

*Azo dyestuffs dyeing on mordants.* C. Jagerspacher, Assr. to Soc. of Chem. Ind. in Basle. U.S.P. 1,375,701, 26.4.21. Appl., 15.11.19.

SEE E.P. 149,553 of 1919; J., 1920, 816 A.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Plant colloids. VIII. Some cellulose dextrans.* M. Samec and J. Matula. Kolloid-Chem. Beih., 1921, 11, 37—73.

SULPHITE-CELLULOSE is not completely dissolved by 50% sulphuric acid, whilst 86% sulphuric acid chars it. Solutions of cellulose in sulphuric acid (50—85%) are at first colourless, but on keeping they speedily become coloured. As the degradation of the cellulose becomes greater the coloration produced by iodine changes from blue to violet, then to various shades of brown, and finally disappears; the relative viscosity rises to a maximum and then falls as the swelling and solution increase. In the degradation, dextrans are produced, some of which are soluble in water whilst others are insoluble. The insoluble products are destroyed by sulphuric acid (60% and above) within the first half hour. As the degradation proceeds the following changes are observed successively:—the dextrin esters are gelatinised by alcohol and the dextrin is precipitated; the dextrin esters and the dextrans are both insoluble in alcohol; the dextrin esters are soluble, the dextrans are insoluble in alcohol; both dextrin esters and dextrans are soluble in alcohol. Phosphoric acid of a greater concentration than 75% at ordinary temperatures forms a colourless, very viscous jelly with cellulose which is only slowly changed at this temperature, but at higher temperatures a

slow degradation occurs and eventually the solution becomes brown or black. The action of sulphuric acid on cellulose depends not only on the concentration of the acid but also on the relative quantities of acid and cellulose. With a sufficient excess of acid dialysable dextrans are produced in the first quarter of an hour and at the same time the molecular weight of the colloidal residue is reduced. The degradation of both dialysable and non-dialysable portions proceeds further by continued action of the acid until after 5 hours the mean molecular weight of the most coarsely disperse portion is 2400. At the same time the  $-SO_3H$  group enters the molecule with the formation of esters. Diastase is capable of converting some of the dextrans produced into fermentable sugars. (Cf. J.C.S. June.)

—J. F. S.

*Colophenic acids.* Aschan. See XIII.

#### PATENTS.

*Wool-washing machines.* J. F. and W. H. White. E.P. 162,313, 8.10.19.

THE wool is carried forward over several consecutive hemi-cylindrical perforated surfaces by means of adjustable drums having projecting tangential beaks, whereby it is constantly immersed in the washing liquid. The tangential beaks are so shaped that there is no tendency for the wool to lap around them.—A. J. H.

*Fireproofing material and heat- and cold-retaining coverings; Process of making — and product thereof.* C. S. Hathaway, Assr. to J. A. Locke. U.S.P. 1,374,885, 12.4.21. Appl., 24.3.20.

A FABRIC is impregnated with a composition containing a tar and an hydraulic cement.—H. S. H.

*Sulphate pulp; Manufacture of —.* E. Olsson. U.S.P. 1,375,759, 26.4.21. Appl., 2.4.19.

A DIGESTION liquor containing sodium hydroxide and a larger amount of sodium sulphide is used.—A. J. H.

*Articles obtained from solutions [cellulose and the like]; Process for the production and simultaneous conversion of — into their ultimate forms.* B. Borzykowski. U.S.P. (A) 1,375,823 and (B) 1,375,824, 26.4.21. Appl., 29.5.17 and 19.6.20.

(A) ARTICLES are produced from solutions of viscose by "forming" and "setting" in a non-acid salt solution and are immediately afterwards converted into the final cellulose product. (B) Articles are produced by passing solutions, capable of coagulation, directly into an alkaline bath and are immediately afterwards subjected to an ultimate chemical conversion; e.g., cellulose thread is produced by spinning a solution of cellulose into an alkaline bath, withdrawing the thread and winding it under tension and simultaneously converting it into cellulose hydrate by treatment with sulphuric acid.—A. J. H.

*Fibres; Process for making —.* R. Kron. U.S.P. 1,376,285, 26.4.21. Appl., 18.9.19.

FIBRES which felt together not too densely and thereby produce an absorbent spongy sheet sufficiently strong and durable for application to the manufacture of rough pasteboard, which after saturation with tar or bitumen, is suitable for roofing, linings, etc., are produced (without the use of alkalis or acids) from wood in the form of logs and similar large pieces and from ligneous or strongly silicious plants. These materials are boiled with water or steamed, rolled out, crushed and ground so as to obtain the natural cellulose fibres without deterioration and not weakened by chemical reagents.—A. J. H.

*Paper material coated with leaf metal.* E. Tscheike, Assr. to The Chemical Foundation, Inc. U.S.P. 1,376,737, 3.5.21. Appl., 6.6.17.

SEE E.P. 113,297 of 1917; J., 1918, 179 A

*Separating solid particles from suspension [e.g., fibres from paper mill waste waters]; Process for —.* N. Pedersen. U.S.P. 1,376,459, 3.5.21. Appl., 3.9.18.

SEE E.P. 119,028 of 1918; J., 1919, 760 A.

*Chlorate explosives.* G.P. 307,100. See XXII.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Anilides of  $\beta$ -[hydr]oxynaphthoic acid [in production of ice colours].* E. R. Brunskill. J. Ind. Eng. Chem., 1921, 13, 309.

ALTHOUGH the colours obtained by substituting  $\beta$ -hydroxynaphthoic acid for  $\beta$ -naphthol in the ice process are brighter, they are not fast to washing; to overcome this defect amides of the acid have been used, the chief of which is the anilide (Naphthol AS). Dyeing experiments were made with the following substances as naphthols:— $\beta$ -hydroxynaphthoic acid, the anilide, the toluidide, and the *p*-chloroanilide; each naphthol was coupled with aniline, *p*-nitroaniline, *p*-chloroaniline-*o*-sulphonic acid, *p*-toluidine, *m*-nitro-*p*-toluidine and *o*-chloro-*p*-toluidinesulphonic acid. All the colours made from  $\beta$ -hydroxynaphthoic acid washed out, but the anilides gave fairly fast colours, except in the case of the sulphonated amides. The latter, however, were rendered fast by immersing the dyed fabric for 30 mins. in 3% calcium chloride solution at 50° C.—W. P. S.

#### PATENTS.

*Dyeing; Method of —.* A. Lendle, Assr. to Kuntroff, Pickhardt and Co. U.S.P. 1,375,919, 26.4.21. Appl., 9.9.20.

FABRIC is dyed with a basic dyestuff and is subsequently treated with a complex acid containing phosphorus and tungsten. (Cf. E.P. 143,242; J., 1921, 385 A.)—A. J. H.

*Decomposition products of proteins [protalbinic and lysalbinic acids]; Use of — [in dyeing].* C. Bennett. G.P. (A) 331,484, 25.12.15, and (B) 332,476, 19.1.16. Addns. to 330,133 and 331,234 (J., 1921, 256 A, 297 A).

(A) The use of the decomposition products, prepared, e.g., by heating casein or gluten with sodium hydroxide, is claimed in weakly alkaline, neutral, or weakly acid vats, but not in strongly alkaline vats. (B) In the case of sulphur dyes suitable for dyeing animal fibres, the vat is prepared by treating the dye with hydrosulphite and sodium hydroxide, and the alkali is neutralised by the addition of an acid or a neutral salt of a volatile alkali and an organic acid, e.g., ammonium formate, together with the protalbinic and lysalbinic acids.—L. A. C.

*Hosiery, gloves, and the like dyed with Aniline Black; Manufacture of —.* K. Schlatter. G.P. 332,675, 23.3.19. Conv., 21.11.18.

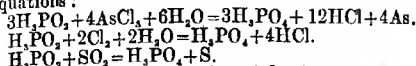
THE goods are made from raw yarn of which a part has been treated with a substance which prevents the injurious effect of the Aniline Black process (such as a metal oxide which can be printed on to the yarn, or a compound capable of combining with an acid or acting as a reducing agent) or of which a part has not been aged. A suitable method of manufacture is to employ double threads, in which one has been dyed with Aniline Black, and the other has been dyed with another black dye, or has been treated as described above.—L. A. C.



## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Hydrochloric acid; New method of purification of*  
— J. Lamquet. Bull. Soc. Chim. Belg., 1920,  
29, 309.

COMMERCIAL hydrochloric acid may be freed from arsenic, chlorine, and sulphur dioxide by the simple addition of hypophosphorous acid or one of its salts, the reactions being represented by the following equations:



By using the barium salt sulphuric acid is precipitated at the same time.—G. F. M.

*Glauber's salt; Manufacture of* — W. Hüttner. Chem.-Zeit., 1921, 45, 313—315.

THE waste product derived from the manufacture of potassium chloride and consisting chiefly of a mixture of rock salt and kieserite, may be utilised as raw material for the production of Glauber's salt. It contains approximately: KCl, 2.6%; MgSO<sub>4</sub>, 10.5%; CaSO<sub>4</sub>, 3.4%; NaCl, 54.3%; and Na<sub>2</sub>SO<sub>4</sub>, 6.9%. The waste heaps are sprinkled with water at 45°–50° C., until a solution is obtained of sp. gr. 1.27–1.29 and of the approximate composition: NaCl, 21.1%; MgSO<sub>4</sub>, 9.1%; MgCl<sub>2</sub>, 1.3%; H<sub>2</sub>O, 68.4%. Formerly it was usual to crystallise this solution in winter at below 0° C., but artificial refrigeration is now adopted, the solution being placed in vats with cooling coils, and depositing fine crystals of Glauber's salt on cooling. The crystals are re-dissolved in steam-heated vessels and freed from iron by precipitation with lime; the solution is then allowed to crystallise again in such a manner as to yield either large crystals or a fine crystalline material, as required. To obtain a calcined product, suitable for glass works, the salt solution is evaporated in a vessel provided with copper heating coils, and the residue is dehydrated.—W. J. W.

*Ammonia and carbonic acid; Compounds of — in equilibrium with their aqueous solutions.* E. Terres and H. Weiser. Z. Elektrochem., 1921, 27, 177—193.

THE conditions under which the different compounds of ammonia and carbonic acid are stable in contact with aqueous solutions of the components were studied between 0.1° and 60° C. Five different compounds can exist as solid phases, namely ammonium bicarbonate, the bicarbonate-carbonate double salt, 2NH<sub>4</sub>HCO<sub>3</sub>·(NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>·H<sub>2</sub>O, the normal carbonate, ammonium carbamate, and the carbamate-bicarbonate double salt, NH<sub>4</sub>CO<sub>2</sub>NH<sub>4</sub>·NH<sub>4</sub>HCO<sub>3</sub>. All these have distinctive crystalline forms by which they can be identified microscopically. Previously only the bicarbonate and carbamate had been prepared in the pure state, but methods have now been devised for preparing the bicarbonate-carbonate double salt and the normal carbonate. The former salt crystallises from solutions containing NH<sub>3</sub> and CO<sub>2</sub> in the ratio 4:5. The normal carbonate is prepared by dissolving 395 g. of ammonium bicarbonate in 150 g. of water and 333 g. of 25% ammonia solution whilst passing in ammonia under a pressure of 0.2 atm. and warming to 40° C. until solution is complete. On cooling to 10° C. the normal carbonate crystallises out. Only the bicarbonate forms congruent solutions, in which the ratio of NH<sub>3</sub> to CO<sub>2</sub> is the same as in the solid phase. The other compounds form stable solid phases only in presence of excess of ammonia. The equilibrium diagram shows that the normal carbonate and the two double salts have a limited temperature range of stability, whilst the bicarbonate and carbamate can exist at all temperatures between 0° and 60° C. with the necessary am-

monia concentration. Starting with bicarbonate and water, between 0° and 33° C., as the concentration of ammonia increases the solid phase changes consecutively to bicarbonate-carbonate, normal carbonate, and finally carbamate. At about 33° C. both the double salt and normal carbonate disappear and the new double salt bicarbonate-carbamate appears between the bicarbonate and carbamate. No further change takes place up to 60° C.

—E. H. R.

*Potassium, sodium, ammonium, and aniline bisulphates, potassium binoxalate and tetroxalate; Decomposition of — by solvents.* T. Sabalitschka and H. Schrader. Ber. deuts. Pharm. Ges., 1921, 31, 183—198.

THE crystallisation of aqueous solutions of potassium, sodium, ammonium, and aniline bisulphates, results in the formation, to a greater or lesser degree, of the normal salt and the free acid. Thus pure potassium sulphate was deposited from a solution of pure potassium bisulphate, whilst a mixture of the normal and acid salts was obtained in the other cases. The addition of alcohol to an aqueous solution of sodium bisulphate caused the precipitation of the pure normal salt. The precipitate obtained in a similar way with ammonium bisulphate solution contained 60–70% of the normal salt. The prolonged extraction of the dry bisulphates with alcohol in a Soxhlet apparatus resulted in the removal of acid, leaving a residue of the normal sulphates in the case of the potassium and sodium salts, and a mixture in the case of the ammonium and aniline salts. The extraction of potassium tetroxalate with ether and alcohol resulted in a similar decomposition into free acid and potassium binoxalate, the latter being unchanged by further action of the solvents.—G. F. M.

*Ammonium sulphate; Melting point of —.* J. Kendall and A. W. Davidson. J. Ind. Eng. Chem., 1921, 13, 303—304.

DIVERGENT figures for the melting point of ammonium sulphate must be ascribed to the instability of the normal salt (cf. Watson Smith, J., 1895, 629). Results obtained by many observers for the melting point of the acid salt are in close agreement with Kendall and Landon's figure, 146.9° C. (J. Amer. Chem. Soc., 1920, 42, 2131), and a melting point of 251° C. given by Jänecke (J., 1920, 818 A), is explicable. The actual melting point of the normal salt can be determined only by heating it in a sealed tube, with no free air space, so as to avoid appreciable loss of ammonia. Under these conditions the salt softened at 490° C. and its melting point was found to be 513° C. ± 2°, under an ammonia pressure of considerably more than 1 atm. —W. J. W.

*Bleaching powder; Solution of —.* M. Champion. Bull. Soc. Chim. Belg., 1920, 29, 24—28.

MIXTURES of bleaching powder and water containing from 1 to 20% of the former were shaken for 3 hours, allowed to settle, and in the clear supernatant solution the active oxygen, total chlorine and calcium, and the density were determined. If the concentration exceeds 4% there is a risk of some of the active oxygen being retained in the residual sludge from the preparation. The figures on the whole show rather higher proportions of calcium and total chlorine to active oxygen than those required by the formula, Cl<sub>2</sub>CaOCl.—W. G.

*Reaction CaCO<sub>3</sub> = CO<sub>2</sub> + CaO; Reversibility of the*  
— P. Jolibois and Bourvier. Comptes rend., 1921, 172, 1182—1183.

USING various forms of calcium carbonate, it was found that the reversal of the dissociation on cool-



ing was never complete except when precipitated calcium carbonate was dissociated by heat in the presence of an excess of calcium oxide.—W. G.

*Calcium carbide; Apparatus and method for estimation of — for calcium cyanamide manufacture.* E. von Drathen. Chem.-Zeit., 1921, 45, 447.

TWENTY grms. of carbide is placed in a stoppered separating funnel, the tap of which has a conical bore, varying in diameter from 1.5 mm. to 2.5 mm., so that when it becomes choked the particles are easily dislodged by turning through 180°. The funnel is fixed in a filter-flask containing a saturated solution of salt, and connected by tubing with a gas-measuring flask. This latter has a capacity of 5 l. and is graduated in 50 c.c. divisions; its lower end is continued as a long tube of 800 c.c. capacity graduated in 10 c.c. divisions, and this is in communication with a levelling flask. The volume of the acetylene generated by introduction of the carbide into the salt solution may be read to within 5 c.c.—W. J. W.

*Chrome alum solutions; Action of sodium carbonate on —.* L. Meunier. J. Soc. Leather Trades' Chem., 1921, 5, 103—111.

As a result of Burton's experiments (J., 1921, 19A) the author has carried out further work on the action of sodium carbonate in precipitating chrome alum solutions. The amount of sodium carbonate necessary to bring about the commencement of precipitation is less when the solution of chrome alum is more dilute, when the temperature is higher, when the solution is old, and when it is stirred vigorously. A reversible change takes place in chrome alum solutions which have been heated for some time at a constant temperature, but the change is irreversible if the heating has been prolonged or has taken place at 100° C. or above. Sodium carbonate precipitates a basic chromium sulphate from chrome alum solutions heated to 100° C. and chromium hydroxide from cold or freshly prepared solutions. Chrome alum solutions undergo hydrolysis and the basic chromic salts so formed polymerise under the action of heat and more slowly on keeping. Precipitation is due to the sodium carbonate acting as a neutralising agent and also as a coagulating agent on the chromium hydroxide or the polymerised basic chromium salts in colloidal solution.—D. W.

*Phosgene; Solvents for —.* C. Baskerville and P. W. Cohen. J. Ind. Eng. Chem., 1921, 13, 333—334.

ONE part by weight of phosgene (carbonyl chloride) is dissolved by the following weights of different solvents at 20°–21° C.:—Carbon tetrachloride, 3.6; chloroform, 1.7; gasoline, 1.2; paraffin oil, not soluble; Russian mineral oil, 2.8; benzene, 1; toluene, 1.5; glacial acetic acid, 1.6; ethyl acetate, 1; chlorocane, 3.2. The last substance is paraffin which has been melted and treated with chlorine. In the case of gasoline, chloroform, and glacial acetic acid reaction was evident by heat of solution and change in h.p. of the solvent; there was also a change in the b.p. of the toluene, but the other solvents appeared to be inert towards the gas.

—W. P. S.

*Sodium-oxygen cell.* Baur. See XI.

*Oxidation of sulphur.* MacIntire and others. See XVI.

*Titration of boric acid.* Boeseken and Couvert. See XVII.

#### PATENTS.

*Nitric acid; Manufacture of concentrated —.* Bayerische A.-G. f. Chem. und Landwirtschaftl. Fabrikate, H. Hackl, and H. Bunzel. G.P. 307,601, 9.3.18.

BARIUM nitrate is heated with sulphuric acid in a vessel through which the mixture is conveyed by a screw device in order to allow of a continuous process.—W. J. W.

*Protection of walls of enclosures in which reactions [e.g., synthesis of ammonia] take place under high temperatures and pressures.* "L'Air Liquide," Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude. E.P. 140,083, 9.3.20, 18.12.18.

THE chamber in which the reaction takes place, being incapable of resisting a very high pressure, is enclosed in a pressure-resisting outer vessel which must be protected from the reacting substances (e.g., gaseous hydrogen). The vessel is therefore filled to a level above the zone of reaction with a liquid that is neutral to the reacting substances and a bad conductor of heat (e.g., potash-soda mixture). To prevent loss of heat by convection the space between the reaction chamber and the outer walls is filled with overlapping rings made of a solid which is also a bad conductor of heat and preferably may be capable of removing any corrosive substance dissolved in the liquid (e.g., rings of magnetic oxide of iron will absorb hydrogen with formation of water vapour, which in turn may be absorbed by metallic iron placed inside the vessel hut above the zone of reaction and out of the intense heat).—B. M. V.

*Aluminium chloride; Production of —.* M. Shoeld, Assr. to Armour Fertilizer Works. U.S.P. 1,375,116, 19.4.21. Appl., 5.11.20.

ALUMINA is heated to 1600°–2000° C. to render it anhydrous, and is then subjected to the action of chlorine, in presence of carbon.—W. J. W.

*Perborates; Manufacture of —.* J. K. Langhard, Assr. to Frederikstad Elektrokem. Fabr. A./S. U.S.P. 1,375,596, 19.4.21. Appl., 6.2.20.

A SOLUTION of a borate is electrolysed in presence of a cyanogen compound.—C. I.

*Per-salts and metallic peroxides; Process for rendering — stable by coating them with gelatin or glue.* H. E. Bergmann. G.P. 334,868, 1.5.19.

THE gelatin or glue solution is first oxidised to destroy reducing impurities and thus avoid loss of active oxygen in the compound to be preserved. The stabiliser may be applied to the reagents before preparation of the peroxides etc., as it does not interfere with the reaction.—C. I.

*Arsine, phosphine, and stibine; Process and apparatus for electrolytic generation of —.* H. Blumenberg, jun. U.S.P. 1,375,819, 26.4.21. Appl., 11.6.19.

A COMPOUND containing phosphorus, or other element of the phosphorus group (nitrogen and boron excepted), is electrolysed in conjunction with a reagent capable of forming nascent hydrogen at the cathode and oxygen at the anode, and the gases formed are collected.—A. J. H.

*Basic zirconium sulphate; Process of making —.* E. J. Pugh, Assr. to Pennsylvania Salt Manufacturing Co. U.S.P. 1,376,161, 26.4.21. Appl., 30.1.19.

CHLORINE is passed over the heated ore and the sublimates of zirconium chloride dissolved in water. Basic zirconium sulphate is precipitated from this solution by addition of sulphuric acid.—A. R. P.

*Magnesium bisulphite solution and magnesium oxide; Process of making* — P. and A. Frank and M. Lehram. G.P. 302,472, 8.9.15.

MAGNESIUM sulphate is converted into oxide by heating with sawdust or other carbonaceous material, and half of the oxide is transformed into bisulphite by saturating with the sulphur dioxide evolved. Excess carbonaceous matter is burnt off. —C. I.

*Alumina; Process for the preparation of* — suitable for the manufacture of aluminium. M. Buchner. G.P. 303,923, 10.9.16.

THE crude ore is decomposed with sulphuric acid, freed from iron, and the purified aluminium salt treated with enough ammonia to precipitate basic sulphate. The precipitate is washed, dried, and ignited in a current of air, the sulphur trioxide and ammonia evolved being recovered. The alumina produced is granular and not powdery. —C. I.

*Nitrogen; Process for the oxidation of* —. Gewerkschaft des Steinkohlenbergwerks Lothringen, and M. Keltling. G.P. (A) 329,846 and (B) 329,847, 17.12.19.

(A) IN a process for the oxidation of nitrogen heated to over 1500° C., the mixture of oxides of nitrogen from the reaction vessel is passed at a high speed (at least 5 m. per sec.) through a preheater consisting of narrow tubes of zirconia. In the reaction vessel a contact body is arranged, the active surface of which is several times that of the catalytic preheater surface. (B) The entering gases are passed through a heating chamber with refractory lining connected with a number of pipes, the front portions of which are insulated. After undergoing the reaction the gases pass on through pipes constricted to a small diameter into a cooling chamber, which may itself be formed of pipes set at right angles to the direction of the current of gas. —C. I.

*Titanium tetrachloride; Process for manufacturing* —. O. Priesz. G.P. (A) 334,248 and (B) 334,249, 10.10.17.

(A) IN the manufacture of titanium tetrachloride by passing chlorine over a heated mixture of titanic acid and charcoal, the activity of the chlorine is increased, and the reaction temperature reduced to below 300° C., by adding a small amount of an oxide or chloride of a rare earth metal, or of a metal of the manganese group, e.g. cerium, zirconium, or manganese. (B) In the treatment of titanic acid by heating it with a mixture of carbon monoxide and chlorine, the addition of small amounts of carbon to the titanic acid increases the yield of titanium tetrachloride. —W. J. W.

*Magnesium hypobromite; Preparation of insoluble basic* —. E. Merck and W. Pip. G.P. 334,654, 12.6.19. Addn. to 297,874.

A MIXTURE of a solution of a magnesium salt and an alkali or alkaline-earth hydroxide is treated with an equivalent weight of hypobromous acid or the corresponding amount of liquid or gaseous bromine. The basic hypobromite is deposited in a gelatinous state and dries to a yellow powder almost insoluble in water. —C. I.

*Sulphur; Treatment of mined* —. R. F. Bacon and H. S. Davis, Assrs. to Texas Gulf Sulphur Co. U.S.P. (A) 1,374,897 and (B) 1,374,898, 19.4.21. Appl., 6.10.19.

(A) THE free burning quality of mined sulphur containing oil is improved by heating it to 400°–430° C. for a sufficient time to carbonise the oil. (B) Crude sulphur containing oil is refined by heating it to carbonise the oil, then allowing the mass

to cool to a temperature above the melting point of sulphur and stand until the carbon has segregated. —A. R. P.

*Sulphur; Process for recovering* — from sulphates, especially calcium sulphate. Metallbank und Metallurgische Ges. A.-G. G.P. 334,247, 23.12.16.

IN calcining a mixture of sulphate and fuel, the depth of the layer of the material and the introduction of air are so regulated that the surface of the material retains a dark appearance during the process. By this means the greater part of the combined sulphur is recovered in the free state. —W. J. W.

*Caustic soda or soda lye; Production of* —. Schweizerische Sodafabrik. E.P. 144,266, 17.5.20. Conv., 4.6.19.

SEE G.P. 332,003 of 1919; J., 1921, 301 A.

*Ammonium sulphate; Method of discharging* — from saturator baths. F. J. Collin, A.-G. zur Verwertung von Brennstoffen und Metallen. E.P. 145,781, 2.7.20. Conv., 2.4.19.

SEE G.P. 316,596 of 1919; J., 1920, 405 A.

*Aluminium hydroxide; Process for the production of* —. M. Buchner. E.P. 162,303, 23.11.17.

SEE U.S.P. 1,337,192 of 1920; J., 1920, 488 A.

*Alkali aluminates; Preparation of pure* —. P. Miguet, Assr. to Rochette Frères. U.S.P. 1,376,563, 3.5.21. Appl., 3.11.19.

SEE E.P. 141,666 of 1919; J., 1920, 748 A.

*Hydrogen; Purification of* —. E. K. Rideal and H. S. Taylor. U.S.P. 1,375,932, 26.4.21. Appl., 11.2.19.

SEE E.P. 129,743 of 1918; J., 1919, 632 A.

*Ammonia from gases.* E.P. 162,314. See IIa.

*Ammonia recovery.* U.S.P. 1,375,475–8 and 1,375,483–5. See IIa.

## VIII.—GLASS; CERAMICS.

*Glass; Dissolved gases in* —. E. W. Washburn, F. F. Footitt, and E. N. Bunting. Univ. Illinois Bull. 15, Vol. XVIII, 13.12.20. 32 pages.

THE percentage of gases dissolved in various samples of glass was determined by melting the sample under atmospheric pressure in an electric resistance furnace connected by means of a valve with a large evacuated tank with an internal pressure of less than 1 cm. and sometimes 0.02 mm. of mercury. When the glass was at a suitable temperature the valve was opened quickly, thus causing a sudden drop of pressure in the furnace and causing the glass to effervesce vigorously and to expand rapidly to about six times its original volume. In a modified form of the apparatus, the furnace consisted of a porcelain tube 3 cm. diam. and 13 cm. high, wound with platinum wire, surrounded by a tightly fitting porcelain protection tube, then by a second porcelain tube, the whole being enclosed in glass. The sample weighing about 25 g. was heated *in vacuo* to 1400° C. until no more gas was evolved. A barium flint optical glass with a refractive index of 1.605 yielded 15.3 c.c. of gas per 50 g. of glass, or 110% by vol. Two other samples yielded 50% by vol. of gas; the gas contained 25% CO<sub>2</sub> and 75% O<sub>2</sub>. A light flint bulb glass gave 20% by vol. of gas consisting of CO<sub>2</sub>, 58%, O<sub>2</sub>, 24%, and N<sub>2</sub>, 18%. A borosilicate glass gave 20% by vol. of gas consisting of CO<sub>2</sub>, 26%, O<sub>2</sub>, 37%, and N<sub>2</sub>, 37%. The amount and

composition of the gas varied with the type of glass and the conditions of melting and planing. The results obtained indicate that if glass were melted on an industrial scale in a vacuum furnace, the customary planing would be eliminated, thus reducing the temperature necessary for finishing the glass, and the yield of perfect glass would be increased owing to the absence of all "seed." (*Cf.* J.C.S., June.)—A. B. S.

## PATENTS.

*Furnaces for melting glass.* The E. W. Harvey Gas Co., and E. W. Harvey. E.P. 162,232, 16.9.20.

HEATING of the glass near gathering or tapping openings of gas-fired glass-melting furnaces with one or more reversible flames is secured by providing supplementary flues in suitable positions near these openings. The flues, which are regulated by dampers, are connected directly with the chimney, and so have a stronger draught than that in the main ports or waste gas flues, these being only indirectly connected with the chimney through regenerators. The method may be applied to various types of tank and pot furnaces.—A. C.

*Quartz glass; Process for making articles of —.* J. Scharl, Assr. to General Ceramics Co. U.S.P. 1,375,657, 19.4.21. Appl., 8.2.19.

QUARTZ glass is fused to a plastic mass in an electric furnace provided with a resistance core. The fused mass is removed from the furnace together with the hot core and blown into the desired shape.

—J. S. G. T.

*Dental cements; Manufacture of —.* S. Schiff. E.P. 161,868, 16.6.20. Addn. to 145,052.

In the manufacture of dental cements of the kind where a powder is mixed with a liquid to produce a composition which hardens to a solid cement, colloidal silica is added for the purpose of increasing the hardness and transparency of the product.

—H. S. H.

*Bricks, slabs, and the like; Method for treating clayey material for making —.* C. W. Wallace. E.P. 162,483, 11.3.20.

THE bricks etc. are formed from the clayey material and passed directly into an ordinary working furnace, where they are well heated throughout by direct contact with the flames of the furnace. They are then withdrawn from the furnace and cooled suddenly by plunging them totally into water. A dense compact brick is thus formed from clayey materials (e.g., certain African clayey materials similar to kaolin) which, if prepared and fired in the usual manner, give products which are too soft and porous to be of real service.—H. S. H.

*Graphite crucibles; Method of making —.* Method of making clay and graphite crucibles. J. H. L. De Bats, Assr. to Lava Crucible Co. of Pittsburgh. U.S.P. (a) 1,374,909 and (a) 1,374,910, 19.4.21. Appl., 31.5.18. Renewed 5.8.20.

(A) NON-FERROUS metal is mixed with the material, containing clay, from which the crucible is made, giving, in use, a surface coating of metallic silicate. (B) By mixing finely-divided metal of the aluminium-magnesium group with material containing clay used for making crucibles, the latter may be burned shortly after moulding.—A. C.

*Highly-refractory article, and method of producing same.* H. H. Buckman and G. A. Pritchard, Assrs. to Buckman and Pritchard, Inc. U.S.P. 1,375,077, 19.4.21. Appl., 9.10.19.

ZIRCON is added to the batch for making refractory material.—A. C.

*Refractory material.* M. L. Hartmann, Assr. to The Carborundum Co. U.S.P. 1,376,091, 26.4.21. Appl., 17.5.20.

A REFRACTORY article contains carborundum and a bonding material containing mixtures of zirconium and aluminium silicates.—H. S. H.

*Brick-kiln.* F. Munhollon. U.S.P. 1,375,719, 26.4.21. Appl., 29.12.19.

A BRICK kiln has a series of furnaces spaced about the outer wall. Transverse passages run beneath the floor of the kiln, and are connected by other passages with the furnaces. The openings into the transverse passages vary in size according to their distance from the furnaces.—H. S. H.

*Annealing furnaces for glass-ware and the like.* H. M. Thompson. E.P. 140,817, 26.3.20. Conv., 1.6.18.

SEE U.S.P. 1,281,713 of 1918; J., 1919, 16 A.

## IX.—BUILDING MATERIALS.

## PATENTS.

*Porous cement or mortar products; Manufacture of —.* A. Sinding-Larsen. E.P. 146,172, 25.6.20. Conv., 3.7.19.

A SUBSTANCE (e.g., aluminium powder), which is capable of reacting with water and alkaline solutions to form a non-volatile compound and hydrogen, is mixed with Portland cement or other mortar. The large volume of hydrogen evolved causes the product to be very porous. It is of advantage to mix the substances in a dry state and to use hot water or a solution of caustic alkali.—H. S. H.

*Stone-like materials of foam-like structure; Method for manufacturing —.* E. C. Bayer. E.P. 162,318, 17.11.19.

STONE-LIKE material permeated with air-filled pores is made by mixing together diatomaceous earth and powdered felspar or rocks containing felspar, forming the mixture into the desired shape with the addition of a little water, and heating to the fusing point of the mixture (about 1200°–1300° C.). The particles of air contained in the diatomaceous earth expand under this treatment and are surrounded by softened or fused solid matter which forms dense walls on cooling.—H. S. H.

*Slag; Process for reducing fused — to granular form.* M. Maguet. E.P. 162,375, 26.1.20.

A JET of highly-heated basic slag from a blast furnace is divided into drops by means of an air blast directed against it. The temperature of the slag is thereby lowered to about 1300° C. On dropping into water the slag forms granules suitable for use in the manufacture of slag cements and bricks.—H. S. H.

*Plastic composition for producing a jointless floor covering.* M. Roberts. E.P. 162,514, 6.4.20.

A PLASTIC composition is produced by incorporating sawdust, cork-dust, or wood pulp, a solution of magnesium chloride, magnesite, and French chalk, with or without colouring matter, and subsequently mixing with a solution of magnesium chloride in water until the desired plasticity is attained.—H. S. H.

*Wood protection; Method of —.* P. Bartsch. U.S.P. 1,374,806, 12.4.21. Appl., 23.10.20.

WOOD is protected from destructive organisms by removing the resin by means of a solvent, displacing the solvent by a chemical which in turn is displaced by an aqueous solution of arsenic and copper, then chemically dehydrating the wood to cause precipi-

tation of poisonous substances, displacing the dehydrant by a paraffin solvent, and finally displacing the latter by poisoned paraffin.—H. S. H.

*Asphalt substitute.* G.P. 332,888. See III.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Electrolytic iron [; Hardness on quenching of —].* O. Bauer and W. Schneider. Stahl u. Eisen, 1921, 41, 647–648.

TEST-PIECES of electrolytic iron, previously annealed at 950° C. and cooled in air, were quenched from different temperatures varying from 650° to 1200° C. The Brinell hardness showed little variation as did also the ultimate breaking stress. Similar results were obtained with iron annealed at 950° C. in *vacuo* and quenched at temperatures varying from 620° to 1100° C. The different results recorded by Boynton (J., 1906, 538) are probably due to the different methods employed for determining hardness.—J. W. D.

*Copper in molybdenum ores; Behaviour of —.* J. P. Bouardi and M. Shapiro. Chem. and Met. Eng., 1921, 24, 847–850.

MOLYBDENUM interferes in the usual iodometric copper assay as it also liberates iodine from potassium iodide. Previous precipitation of the copper by zinc or aluminium is incomplete in the presence of molybdenum, but the latter may be removed by prolonged boiling of the solution with caustic soda. Double precipitation is, however, necessary to eliminate all the molybdenum, and the following modification of the thiocyanate method is, therefore, recommended. 0.5–5.0 g of finely powdered ore is dissolved in 10–15 c.c. of nitric acid, 5–10 c.c. of hydrochloric acid is added, the mixture evaporated nearly to dryness, the mass treated with a little water, 10–15 c.c. of dilute sulphuric acid (1:1) added, and the whole evaporated till fumes appear. The mass is cooled, treated with water, and the solution filtered. The filtrate is diluted to 250 c.c., treated with ammonia till just alkaline, then with 2 c.c. of sulphuric acid (1:1) and 2–3 g. of anhydrous sodium sulphite. To the boiling solution is then added 10–25 c.c. of a 20% ammonium thiocyanate solution, and the resulting copper precipitate is filtered off and washed till free from the blue colour of the reduced molybdenum compounds. It is redissolved in nitric acid, the solution evaporated nearly to dryness, and the assay finished by the iodide method as usual. Alternatively, the precipitate of cuprous thiocyanate may be extracted with hot caustic soda, the cuprous hydroxide filtered off, and the filtrate acidified and titrated with permanganate. In this case a weighed amount of pure copper should be put through the whole process as a standard. The separation of copper minerals from those containing molybdenum is briefly discussed.—A. R. P.

*Tin in cassiterite; Determination of —.* H. Corti. Anal. Assoc. Quím. Argentina, 1921, 9, 44–53.

THE method proposed is a modification of that of Fresenius. Half a gram of the powdered material is mixed with 5 g. of a mixture of sodium carbonate and sulphur and placed in a crucible, in the bottom of which is about 2 g. of sodium carbonate. A covering of the sodium carbonate-sulphur mixture is added and the crucible placed in a cold muffle furnace. The muffle is heated to redness and maintained thereat for 2 hrs. After cooling, the melt is extracted with water and the residue washed with weak ammonium sulphide. Filtrate and washings

are nited and stannic sulphide is precipitated by adding 25 c.c. of hydrochloric acid (1:4). The stannic sulphide is collected on a filter and washed with 1% hydrochloric acid. It is then dissolved in strong hydrochloric acid with gentle boiling, the last traces of hydrogen sulphide being removed by the addition of a few drops of bromine. Ammonia is added till the reaction is alkaline and the liquid raised to boiling. The precipitate of hydrated stannic oxide is washed by decantation, using 1% ammonium nitrate containing a little ammonia, collected, dried, ignited, and weighed as stannic oxide.—G. W. R.

*Bismuth [; Determination of —] in high-grade ores.* G. Spurge. Chem. Age, 1921, 4, 584.

THE following colorimetric method is recommended as giving in 15 mins. results correct to 0.2–0.3% in ores containing up to 45% Bi. 0.05 g. of finely powdered ore is dissolved in 100 c.c. of nitric acid (1:3) and the solution diluted to 500 c.c. A portion is filtered through a dry paper and transferred to a 25 c.c. burette, while a second burette contains a standard solution of bismuth (1 c.c. = 0.00005 g. Bi). Two Nessler tubes are prepared, each containing 10 c.c. of a 10% potassium iodide solution and 3 drops of a solution of sulphur dioxide. 1 c.c. of the bismuth standard solution is added to one tube and the assay is run into the other until the colours match. Lead and copper in large quantities are the only interfering metals. The former is removed by addition of sulphuric acid before making up to bulk and the latter by filtering off any precipitated iodide before comparing the colours, or by precipitating the bismuth as basic nitrate prior to making up to bulk.—A. R. P.

### PATENTS.

*Steel; Process of making —.* E. T. Pelton. U.S.P. 1,376,160, 26.4.21. Appl., 6.7.20.

A CHARGE of iron materials low in phosphorus and sulphur is melted in an acid-lined furnace, and the slag is removed from the surface of the molten metal, which is then treated with a quantity of iron ore. Ebullition is produced in the mixture and the charge poured.—A. R. P.

*Gases derived from roasting ores; Removal of dust from —.* Manufactures de Prod. Chim. du Nord, Etablissements Kuhlmann. E.P. 147,020, 6.7.20. Conv., 6.5.14.

IN a dust-settling chamber the gas stream is caused to meet a series of chain curtains. The curtains are formed of a number of parallel single chains, each of which is arranged in relation to the adjacent ones so as to divide the gas stream as finely as possible. The separate chains may be connected together by a wire which affords a means of shaking the curtains and causing the collected dust to fall.—C. A. K.

*Slags containing iron; Process of separating — from coke and other fuel residues.* F. Krupp A.-G. Grusonwerk. E.P. 152,642, 23.8.20. Conv., 11.10.19. Addn. to 150,333.

PARTICLES of coke remain attached to the iron after the magnetic separation of slags containing iron as described in the chief patent (J., 1921, 139 A). The magnetic material is therefore ground and subjected to a second magnetic treatment.—C. A. K.

*Ores; Treatment of argentiferous lead-zinc sulphide —.* F. E. Elmore. E.P. 162,026, 18.12.19.

FINELY ground ore or concentrate is mixed with alkali or alkaline-earth chlorides, with or without the addition of zinc chloride, and heated to 450°–500° C. in an oxidising atmosphere. Lead together with a proportion of the silver is obtained in solution by extracting the oxidised mixture with hot brine,

and may be recovered by cooling the liquor. From the zinc sulphide which remains unattacked the remaining portion of the silver may be extracted by treatment with suitable solvents. (See also E.P. 129,773, 135,968, and 141,044; J., 1919, 635 A; 1920, 117 A.)—C. A. K.

*Zinc solutions; Purification of* — S. Field, and The Metals Extraction Corp., Ltd. E.P. 162,030, 19.12.19.

ZINC sulphate solutions acidulated, and at a temperature of 70°–100° C., are treated with either finely divided zinc or aluminium or an aluminium-zinc alloy in the form of powder or granules, in the presence of mercuric sulphate. These metals are more electro-positive than the metallic impurities present and cause the latter to be precipitated upon the mercury in the form of a sludge.—J. W. D.

*Electro-deposition of metals upon iron and alloys of iron* [; *Cleaning process preliminary to* —]. R. J. Fletcher. E.P. 162,391, 28.1.20.

THE iron or iron alloy is cleaned by being used alternately as cathode and anode in an acid bath containing copper sulphate, a current density of 150–500 amps. per sq. ft. being employed.

—J. S. G. T.

*Aluminium alloys.* T. F. Bradbury, and Rolfe-Royce, Ltd. E.P. 162,467, 25.2.20.

ALUMINIUM alloys containing 3–15% Cu, or 5–20% Zn, or 1–20% Zn and 1–6% Cu, show an increased fluidity when molten and reduced tendency to crack during solidification, and give sounder castings. Antimony, magnesium, and titanium may be added between the total limits of 0.5 and 3%, the higher limits of the individual metals being 1.5% Sb or Mg and 0.8% Ti.—C. A. K.

*Alloys; Method of treating light-metal* — W. R. Veazey, Assr. to Dow Chemical Co. U.S.P. 1,374,968, 19.4.21. Appl., 21.1.20.

ALLOYS of high magnesium content are heated to 500°–700° F. (260°–370° C.) and then forged or similarly worked.—J. W. D.

(a) *Acid-resisting alloy.* (b, c) *Alloy.* A. W. Clement, Assr. to The Cleveland Brass Manufacturing Co. U.S.P. (a) 1,375,081, (b) 1,375,082, and (c) 1,375,083, 19.4.21. Appl., (a) 27.7.18, (b, c) 15.8.18.

(a) AN acid-resisting alloy contains approximately 40% Cr and a metal having the properties of molybdenum, together with iron and carbon. (b) The principal components are molybdenum and a metal possessing the properties of nickel and cobalt, together with not more than 4% Mn. (c) Iron not exceeding 10% in quantity replaces the manganese in (b).—C. A. K.

*Acid-resisting alloy. Acid-resisting article and method of making same.* A. W. Clement, Assr. to The Cleveland Brass Manufacturing Co. U.S.P. (a) 1,375,672 and (b) 1,375,673, 26.4.21. Appl., 26.7.17.

(a) AN alloy of 60% Cr, 2–4% Si, 2–5% C as graphite, and the remainder iron. (b) Articles made of iron-chromium alloys free from carbon are made acid-resisting by treating the surfaces in a suitable manner with carbon and then cooling rapidly, whereby iron and chromium carbides are produced in the surface layer.—A. R. P.

*Alloys; Electrical-resistance* — F. F. Hansen, Assr. to Hansen-Halliburton Mfg. Co. U.S.P. (a) 1,375,454 and (b) 1,375,455, 19.4.21. Appl., 19.9.19.

(a) AN alloy containing approximately equal

amounts of chromium and copper and not less than 10% of tungsten and molybdenum together, the tungsten being in excess of the molybdenum. (a) An alloy of chromium, tungsten, and aluminium, the chromium being in preponderating amount compared with the combined amount of tungsten and aluminium, and the tungsten being in excess of the aluminium.—J. W. D.

*Alloy.* C. D. Stovall. U.S.P. 1,375,804, 26.4.21. Appl., 14.7.20.

AN alloy containing Au 72 pts., Ni 13½ pts., and Cu 16½ pts.—C. A. K.

*Metal alloy.* J. F. Wandersee, R. Peretto, and T. A. L. Albrecht, Assrs. to Ford Motor Co. U.S.P. (a) 1,376,056 and (b) 1,376,062, 26.4.21. Appl., 10.9.19 and 7.6.20.

(a) THE alloy consists of 2–5% C, 35–65% Co, and the remainder chromium and tungsten. (b) An alloy of 20–35% Cr, 5–20% W, 1–15% Fe, and the remainder cobalt and carbon.—A. R. P.

*Ores; Treatment [chloridising] of* — J. H. Hirt, Assr. to Alvarado Mining and Milling Co. U.S.P. 1,375,002, 19.4.21. Appl., 24.1.20.

ORES, metallurgical products, and the like are chloridised by heating in a closed multiple-hearth furnace and then introducing a chloridising agent in a gaseous or vapour form.—A. R. P.

*Ores; Separation of mixed sulphide* — C. Faul and H. Lavers, Assrs. to Minerals Separation North American Corp. U.S.P. 1,375,087, 19.4.21. Appl., 12.3.15.

A MIXED sulphide ore is concentrated selectively by means of a froth flotation process in presence of a copper material and an alkali bichromate.

—C. A. K.

*Ore concentration process.* W. A. Scott, Assr. to Minerals Separation, North American Corp. U.S.P. 1,375,233, 19.4.21. Appl., 10.9.17. Renewed 15.9.20.

A gas is introduced into the ore pulp in the form of minute bubbles, the resulting mineral-carrying bubbles are treated to remove the gas from them before they reach the surface of the pulp, and the mineral carried by the bubbles is separated from the remainder of the ore.—A. R. P.

*Sodium; Manufacture of metallic* — A. W. Smith and W. R. Veazey. U.S.P. 1,375,330, 19.4.21. Appl., 16.7.17.

A FUSED mixture of sodium carbonate and another salt is electrolysed, the ingredients being proportioned so that the mixture has a lower melting point than any of its constituents.—J. W. D.

*Metallurgical furnace.* D. Baird, Assr. to General Chemical Co. U.S.P. 1,375,346, 19.4.21. Appl., 17.6.20.

A MULTIPLE-HEARTH roasting furnace is provided with means for causing the material to fall from one hearth to another, and with an air conduit situated behind the stream of falling ore, so that an air blast may be directed into the stream.—A. R. P.

*Furnace; Metallurgical* — J. W. Griswold and H. O. Loebell. U.S.P. 1,375,754, 26.4.21. Appl., 24.7.18.

A CLOSED melting chamber is surrounded by a number of combustion channels in which a mixture of gas and air is burnt. Products of combustion are passed through a detached heat exchanger, and the furnace is mounted on trunnions for pouring.

—C. A. K.

*Converters; Gas-discharge conduit for rotary* —  
W. H. Howard, Assr. to American Smelting and Refining Co. U.S.P. 1,375,965, 26.4.21. Appl., 4.12.19.

THE waste gas discharge flue from the upper side of a horizontal cylindrical matte converter is brought to a position concentrically disposed to the axis of the converter by means of right-angled bends. A loose sleeve connecting the discharge end portion with the flue leading to the chimney permits of rotation of the converter.—C. A. K.

[Mineral] values; Process for the extraction of —  
from highly dilute solutions. M. L. E. Baudin. U.S.P. 1,375,441, 19.4.21. Appl., 7.4.17.

MINERAL compounds are extracted from very dilute solutions by bringing the latter into contact with a support coated with a colloidal absorbent material.—A. R. P.

*Silver-solder.* W. B. Bocchetti. U.S.P. 1,375,552, 19.4.21. Appl., 10.2.20.

A SOLDER containing Ag 55–65%, Cu 20–30%, Zn 10–20%, and arsenic trioxide.—J. W. D.

*Brass scrap; Process of treating* —. O. C. Ralston. U.S.P. 1,375,930, 26.4.21. Appl., 2.1.20.

THE copper content of copper-bearing alloys is increased by treatment of the alloy with an aqueous solution containing a solvent for metals electro-positive to copper.—C. A. K.

*Bearing metal.* J. G. Kelly, A. L. Pringle, and H. Hall. U.S.P. 1,376,339, 26.4.21. Appl., 7.5.20.

THE alloy consists of 1 pt. of aluminium, slightly more than 1 pt. of zinc, 2 pts. of lead, and 4 pts. of tin.—A. R. P.

*Ores or concentrates; Process of roasting* —. P. R. Middleton, Assr. to J. C. Lalor. U.S.P. 1,376,025, 26.4.21. Appl., 20.1.20.

ORE containing a metallic sulphide is heated in presence of air to a temperature below the ignition point of the sulphur, and maintained at this temperature until the maximum proportion of the metallic sulphide has been oxidised to sulphate.—W. J. W.

*Iron; Bessemerising* —. R. S. McCaffery. E.P. 138,900, 10.2.20. Conv., 13.11.18.

SEE U.S.P. 1,338,655 of 1920; J., 1920, 455 A.

*Alloys; Process of producing* — and alloys produced thereby. Rare Metals Reduction Co., Assees. of J. B. Grenagle. E.P. 138,348, 27.1.20. Conv., 7.12.15.

SEE U.S.P. 1,248,648 of 1917; J., 1918, 95 A.

*Tin; Method of recovering* — from stanniferous waste products. T. A. Eklund. E.P. 138,622, 2.2.20. Conv., 1.2.19.

SEE G.P. 316,111 of 1919; J., 1920, 303 A.

*Ferro-silico-magnesium alloy; Method of producing* —. G. Pistor, A. Beielstein, and A. Beck, Assrs. to Chem. Fabr. Griesheim-Elektron. U.S.P. 1,376,113, 26.4.21. Appl., 11.11.16. Renewed 7.2.21.

SEE E.P. 100,848 of 1916; J., 1917, 600.

*Alumina.* G.P. 303,923. See VII.

*Slag.* E.P. 162,375. See IX.

*Stencil plates etc.* U.S.P. 1,376,366–9. See XI.

## XI.—ELECTRO-CHEMISTRY.

*Fuel cells; Construction of high-temperature* —. E. Baur, W. D. Treadwell, and G. Trümpler. Z. Elektrochem., 1921, 27, 199–208.

IN a former paper, fuel cells were described in which copper oxide was used as a cathode (J., 1916, 1266). A number of cells of varying construction have now been devised in which iron oxide is substituted for copper oxide. The cells described are of two types, carbon-oxygen and hydrogen-oxygen, and are all characterised by the use of a porous diaphragm in the form of a tube or crucible of magnesia, or better, of a specially-prepared magnesia-clay mixture suitably moulded and fired. One of the most satisfactory cells described had electrodes respectively of iron and magnetite packed in two channelled magnesia-composition tubes, air being passed through the Fe<sub>2</sub>O<sub>3</sub> tube and hydrogen through the iron tube. The electrolyte, equal parts of fused caustic soda and potash, was contained in an iron vessel, and the surface of the electrolyte was covered with a considerable thickness of loose magnesia, in which the electrode tubes were embedded in a horizontal position. The porosity of the magnesia and of the tubes was sufficient to allow the electrolyte to make good contact with the electrodes. Such a cell gave an E.M.F. at 800° C. of 0.90 volt, and had an internal resistance of about 3 ohms. The opinion is expressed that such fuel cells are a technical possibility.—E. H. R.

*Sodium-oxygen cell.* E. Baur. Z. Elektrochem., 1921, 27, 194–199.

A METHOD has been devised for measuring directly the sodium-oxygen potential in a caustic soda cell at temperatures between 300° and 600° C. A number of different types of electrode were first tested by measuring the hydrogen-oxygen potential in caustic soda between 300° and 400° C. Measurements of the sodium-oxygen potential were eventually made in three types of cell, viz., with an alloy of sodium and lead containing 40 mols. % Na against a silver-oxygen electrode, with pure sodium against the same oxygen electrode, and with pure sodium against an iron-oxygen electrode. The lead-sodium alloy gave results about 0.3 volt lower than pure sodium. The cells containing pure sodium gave a mean result of 2.2 volts at 340° C., which corresponds with the value found by Neumann and Bergve for the decomposition potential of sodium hydroxide (J., 1915, 1088). The temperature coefficient found was, however, smaller than theirs, namely,  $-1.3 \times 10^{-3}$  instead of  $-2.95 \times 10^{-3}$ . Some experiments were also made on the behaviour of the cells when supplying current. As the current increases, polarisation sets in, but may be partly prevented by increasing the rate of the oxygen current.—E. H. R.

## PATENTS.

*Melting and other furnaces; [Packing for] electrically heated* —. The Morgan Crucible Co., and C. W. Speirs. E.P. 162,246, 7.10.19.

IN using electrically heated crucibles etc. of the type described in E.P. 24,626 of 1912 (J., 1913, 1019), mounted in a metal or other casing from which they are insulated by refractory brick, access of air to the carbon resistors is avoided by using natural crystalline plumbago of a "platey" nature as a packing between the resistor and the refractory wall of the furnace. A layer of fine powder, such as sand, is placed at the bottom of the annular space between the resistor and the refractory wall.—J. S. G. T.

*Electric furnace.* W. K. Booth and W. L. Morris, Assrs. to The Booth Electric Furnace Co. U.S.P. 1,376,072, 26.4.21. Appl., 9.4.20.

AN electric furnace having a refractory lining is provided with means for rotation about its axis. Electrodes, extending axially through the end walls of the furnace, are carried by adjustable supports mounted on the end walls and are provided with metal collars through which water may be circulated during rotation of the furnace.—J. S. G. T.

*Electrolytic cell.* H. I. Allen and K. R. Fox, Assrs. to Electron Chemical Co. U.S.P. 1,374,976, 19.4.21. Appl., 1.6.20.

AN anode for use in the electrolysis of saline solutions consists of one or more flat carbon plates provided with spaced parallel ribs, the faces of the ribs and of the plates between the ribs being active.—J. S. G. T.

*Battery electrode; Depolarising — and process of making same.* R. C. Benner and H. F. French, Assrs. to National Carbon Co. U.S.P. 1,375,647, 19.4.21. Appl., 22.8.18.

Cuprous oxide prepared electrolytically is oxidised to cupric oxide and then mixed with sulphur.—J. S. G. T.

*Electrically preparing stencil plates, die plates and the like; Solution or bath for —.* G. E. Wertheimer. U.S.P. (a) 1,376,366, (a) 1,376,367, (c) 1,376,368, and (b) 1,376,369, 26.4.21. Appl., (a) 24.12.17, (b, c, d) 16.5.18.

A SOLUTION for the electrolytic treatment of metallic plates or blocks is composed of an aqueous solution of sulphuric acid and nitric acid together with (A) ammonium chloride, (a) ammonium chloride with a small proportion of copper nitrate, (c) ammonium chloride with a small proportion of acetic acid, (d) small proportions of iodine, copper nitrate, and acetic acid.—J. S. G. T.

See also pages (a) 421, *Electrical gas purification* (E.P. 145,477). 426, *Liquid fuel* (U.S.P. 1,376,180). 432, *Perborates* (U.S.P. 1,375,596); *Arsine, phosphine, and stibine* (U.S.P. 1,375,819). 434, *Quartz glass* (U.S.P. 1,375,657). 436, *Electrodeposition* (E.P. 162,391); *Electrical-resistance alloys* (U.S.P. 1,375,454—5).

## XII.—FATS; OILS; WAXES.

*Herring oil.* T. Lexow. Chem. Umschau, 1921, 28, 85—86, 110—113.

THE following constituents are well established:—Myristic, palmitic, zoomaric, stearic, oleic, clupanodonic, gadoleic, and erucic acids, together with acids of formulæ  $C_{18}H_{34}O_2$  and  $C_{22}H_{40}O_2$ , hydroxylated acids, and unsaponifiable matter. Oil from salted herrings also contains lactones, lipoids, and soaps. Formation of free fatty acids begins the moment the herring is dead; the quantity in ordinary good quality oil lies between 2 and 5%. The colour and flavour show no close relationship to the free fatty acid content, the former depending largely on the fishes' nourishment and the method of pickling. The author finds the saponification value unaffected by the season of the year or the size of the fish. There is, however, a considerable variation of this value according to the latitude in which the fish are caught, average values rising from 184 to 189 with an increase in latitude from 66° to 71°. The percentage of unsaponifiable matter varies in a similar way, but inversely with increase in latitude. About 120 determinations of the iodine value (Wijs) show that this varies markedly with the season of the year, in the same way as the yield of oil—the two latter figures being at their highest

(149 and 13%) in July, August, and September and at their lowest (110 and 2%) in March.—H. C. R.

*Critical temperatures of solution of fats in mixtures of ethyl and amyl alcohols.* A. J. J. Vandeynde. Bull. Soc. Chim. Belg., 1921, 30, 14—16, 58—62.

EXPERIMENTS were undertaken to determine whether a mixture of 94% ethyl alcohol with isoamyl alcohol (cf. J., 1914, 1074), which gave critical solution temperatures similar to those of Crismer's ethyl alcohol, could advantageously replace the latter. A mixture which gave with light petroleum the same CST as Crismer's alcohol was found to give lower values with glycerides, and a mixture which gave with beef fat the same CST as Crismer's alcohol, gave, especially with cacao butter and light petroleum, higher values than those of Crismer's alcohol. The distances between the extreme critical solution temperatures of the substances examined are greater for Crismer's alcohol than for the amyl alcohol mixtures, which gives the former a greater sensitiveness. The divergences observed with the amyl alcohol mixtures seem to be due to the relative solubility of the fats in amyl alcohol.—G. F. M.

*Sulphonated oils; Improved formulae for the calculation of inorganic impurities in —.* R. Hart. J. Amer. Leather Chem. Assoc., 1921, 16, 159—161. (Cf. J., 1921, 226 A.)

THE formula previously cited may be simplified when 2A is equal to or < A<sub>s</sub> since no carbonate can be present.

Inorganic impurities = Ash - 0.0634(2A + A<sub>s</sub>). When 2A > A<sub>s</sub>, sodium carbonate will be found in the ash. By substituting A<sub>s</sub> for 2A in the sulphate term the formula becomes

Inorganic impurities = Ash - (0.0795A<sub>s</sub> + 0.0946A). These new formulae are simplified by substituting S (the percent. combined SO<sub>3</sub>) for its equivalent A<sub>s</sub>. They become in the absence of carbonate

Inorganic impurities = Ash - 0.1268(7S + A), and where carbonate is present

Inorganic impurities = Ash - 0.0946(11.77S + A).

The data required are:—ash, combined SO<sub>3</sub> (S), and fixed alkalinity (A) in mg. of KOH.—D. W.

*Oxidation of vaseline and paraffin.* Langen. See IIa.

## PATENTS.

*Oils and fats; Refining of —.* V. Schwarzkopf. E.P. 138,115, 22.1.20. Conv., 25.10.18.

THE oil is mixed with 4—8% of bleaching earth, e.g., fuller's earth, and heated *in vacuo* to 250° C. in a current of hydrogen until free fatty acids no longer distil over.—H. C. R.

*Hydrogenation of unsaturated oils and fats; Process for the continuous —.* E. R. Bolton. E.P. 162,370, 26.1.20.

THE oil is hydrogenated in vessels fairly tightly packed with nickel wool which has been activated by treatment with nitric acid, roasting, and reducing in a current of hydrogen. The activity of the catalyst may be restored *in situ* by washing with hot oil to remove soaps, then with an oil solvent, and finally re-heating in hydrogen.—H. C. R.

*Hydrogenation of oils, fats, and the like; Method of maintaining the activity of metallic catalysts during the —.* E. R. Bolton and E. J. Lush. E.P. 162,382, 27.1.20.

THE poisoning of the nickel catalyst by soap formation is avoided by the addition of a small quantity of mono- or diglycerides of fatty acids to the oil to be treated. This combines with any free fatty acids present or formed during hydrogenation and so prevents the attack of the nickel. Oils contain-



ing free fatty acids can be hydrogenated directly after adding enough mono- or diglycerides to combine with the free fatty acids.—H. C. R.

*Oils, fats, and fatty acids; Process of extracting* — R. H. McKee. U.S.P. 1,376,211, 26.4.21. Appl., 24.5.19.

THE material containing oil is dehydrated and subjected to the solvent action of liquid sulphur dioxide.—B. M. V.

*Fat; Stimulating the production of — in microbes.* Reichsausschuss für pflanzl. und tier. Oele und Fette, G.m.b.H. G.P. 332,411, 12.10.18.

THE microbes are exposed in the form of a thin layer to the action of air containing alcohols or their derivatives, e.g., air mixed with the gases derived from fermentation processes.—L. A. C.

*Fats and oils; Process for neutralisation [and hydrogenation] of* — H. Schlinck & Co. G.P. 334,659, 15.8.16. Addn. to 315,222 (J., 1920, 198 A).

NEUTRALISATION and hydrogenation are effected simultaneously by adding glycol and a suitable catalyst to the fat or oil and then treating it, at a high temperature, with hydrogen, preferably in excess.—J. H. L.

*Catalytic agents for reduction or hydrogenation purposes; Process for producing* — A. Radisson (Assr. to Soc. de Stearinerie et Savonnerie de Lyon), and P. Berthon. U.S.P. 1,377,153, 3.5.21. Appl., 29.10.19.

SEE E.P. 107,004 of 1917; J., 1918, 431 A.

*Deodorising charcoal.* E.P. 162,117. See IIb.

*Products soluble in or forming emulsions with water.* E.P. 134,223. See III.

*Vulcanised-oil product.* U.S.P. 1,376,172—4. See XIV.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Colophony; New constituents of — colophenic acids and their analogues.* O. Aschan. Ber., 1921, 45, 867—886.

A NEW series of acids,  $C_{20}H_{30}O_4$ , has been isolated from the residues left after the preparation of pinabietic acid from the "black liquor" of sulphate-cellulose factories (J., 1919, 646 A) and also from American and Finnish colophony. These are provisionally termed colophenic acids. They are monobasic acids which are differentiated from the resin acids by the ready solubility of their sodium salts in cold water and the stability of the solutions towards carbon dioxide. The lactone ring and the aldehydic group are not present. The presence of the ketonic group could not be definitely established but one oxygen atom is present in a hydroxyl group. The acids are colourless or pale yellow, whereas solutions of their alkali salts are dark yellow or brownish-yellow.

Hexadecacolophenic acid,  $C_{16}H_{22}O_4$ , m.p. 96°—105° C. (yellow granules, from concentrated solution) or 86°—98° C. (almost colourless powder, from dilute solution),  $[\alpha]_D^{17} = +22.33^\circ$  (colourless form) or  $+21.66^\circ$  (yellow form) in benzene solution, heptadecacolophenic acid, m.p. 100°—105° C. after softening at 70°—75° C., oktoadecacolophenic acid, and eikocolophenic acid, which softens at 80°—90° C. and is molten below 110° C. are described. Hexadecacolophenic acid is prepared artificially by the oxidation of a slightly alkaline solution of pinabietic acid with hydrogen peroxide. (Cf. J.C.S., July.)—H. W.

*Resin of maritime pine; Acid constituents of the* — G. Dupont. Comptes rend., 1921, 172, 1184—1186. (Cf. J., 1921, 357 A.)

PIMARIC acid as isolated from pine resin and purified by Vesterberg's method (cf. J., 1906, 82) consists of a mixture of 37% of d-pimaric acid and 63% of l-pimaric acid.—W. G.

#### PATENTS.

*Paint vehicle. Paint composition. Paint vehicle for anhydrous pigments. Paint composition.* W. N. Blakeman, jun. U.S.P. (A) 1,375,352, (B) 1,375,353, (C) 1,375,354, and (D) 1,375,355, 19.4.21. Appl., 12.6.17.

A MIXTURE of (A) hydrogenated oil, tung oil, and marine animal oil or (C) untreated commercial marine animal oil and tung oil is used as a paint vehicle. As a paint composition (B) a mixture of an anhydrous pigment with the vehicle described under (C), or (D) an anhydrous mixture of pigment with a fluid hydrogenated oil may be used.

—D. F. T.

*Resinous condensation products [from hydrogenated naphthalene]; Manufacture of* — A.-G. für Anilin-Fabr. G.P. 305,575, 19.4.17.

HYDROGENATED naphthalene is treated with formaldehyde in the presence of acids, with or without the addition of a solvent or diluent. The products can be worked up with turpentine and linseed oil for the manufacture of varnishes.—L. A. C.

*Wood; Process for impregnating — before painting.* H. Haarhaus and W. Hoffmann. G.P. 332,908, 3.3.20.

TO accelerate the drying of oil paints when applied to wood, the latter is first impregnated with a neutral or slightly alkaline solution of alkali naphthenates, and then treated with salts of iron, aluminium, zinc, lead, manganese, or cobalt, to cause precipitation of metallic naphthenates.

—W. J. W.

*Asphalt substitute.* G.P. 332,888. See III.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### PATENTS.

*Vulcanising processes and apparatus for the manufacture of high pressure expanded vulcanised rubber and like substances.* C. L. Marshall. E.P. 162,176, 15.4.20.

FOR the manufacture of a semi-rigid expanded vulcanised rubber, ceresin wax or similar material is incorporated into the mixture so as to render it capable of retaining an inert gas under pressure. By enclosing the material before vulcanisation in a skin of rubber which will allow the diffusion of gas, the vulcanised foam is protected against subsequent mechanical disruption. Vulcanisation is effected in an externally heated chamber containing a removable inner chamber which is provided with inlet and outlet valves for the automatic control of the internal gaseous pressure transmitted from the compressed atmosphere of the larger vessel. The froth formation is developed by a rapid partial reduction of pressure at the completion of vulcanisation, whilst the rubber is still hot.—D. F. T.

*Vulcanisation of caoutchouc; Process for the* — S. J. Peachey. E.P. 162,429, 12.2.20. Addn. to 129,826 (J., 1919, 688 A).

IN the vulcanisation of rubber by the action of hydrogen sulphide and sulphur dioxide, the process is accelerated by applying one or both of the gases under pressure.—D. F. T.

*Vulcanised rubber goods; Methods for the preservation of* —. H. P. Stevens. E.P. 162,528, 16.4.20.

THE rate of perishing of vulcanised rubber decreases with the proportion of moisture in the surrounding atmosphere. For the preservation of rubber goods, therefore, the atmosphere of the storage chamber is kept saturated with moisture, or nearly so. (Cf. J., 1920, 251 r.)—D. F. T.

*Vulcanised-oil product.* W. O. Snelling. U.S.P. (A) 1,376,172, (B) 1,376,173, and (C) 1,376,174, 26.4.21. Appl., (A, B) 8.8.19 and (C) 21.8.19.

(A) A VULCANISED oil product is depolymerised in the presence of hydrogen chloride. (B) Various degrees of plasticity are imparted to different portions of vulcanised oil, and the products are mixed. (C) A liquid vulcanised oil product capable of being solidified by contact with water is claimed. (Cf. U.S.P. 1,315,246 of 1919; J., 1920, 697 A.) —D. F. T.

*Rubber; Drying* — by means of indifferent gases. A.-G. Metzeler und Co. G.P. 332,974, 21.2.19.

FLUE gases freed from ashes, soot, sulphur dioxide, etc., and cooled or heated as the case may be, are employed.—J. H. L.

*Rubber-like products; Hindering oxidation in synthetic* —. Badische Anilin- und Soda-Fabr. G.P. 332,305, 28.3.18. Addn. to 330,741 (J., 1921, 312 A).

THE material is mixed with aromatic nitro-compounds, e.g., 1% of nitronaphthalene, nitrobenzene, etc., instead of phenols or naphthols. The products can be vulcanised as usual.—J. H. L.

*Rubber-like products; Preparation of* —. Badische Anilin- und Soda-Fabr. G.P. 333,496, 22.3.18. Addn. to 329,676 (J., 1921, 400 A).

THE polymerisation is carried out in presence of aromatic hydroxy- or nitro-compounds, or other substances which hinder oxidation of synthetic rubber-like products (cf. preceding abstract). The added substances remain homogeneously distributed throughout the products and minimise oxidation during the working of the latter. —J. H. L.

*Rubber products; Manufacture of* —. Goodyear Tire and Rubber Co., Assees. of C. W. Bedford. E.P. 148,992, 6.7.20. Conv., 29.5.17.

SEE U.S.P. 1,323,951 of 1919; J., 1920, 123 A.

*Vulcanising; Method and apparatus [annular moulds heated by induced electric currents] for* —. Goodyear Tire and Rubber Co., Assees. of C. W. Post. E.P. 145,425, 17.6.20. Conv., 22.5.19.

## XV.—LEATHER; BONE; HORN; GLUE.

*Tanning material; Yield of extract from* —. A. Harvey. J. Soc. Leather Trades' Chem., 1921, 5, 115—117.

IN calculating the yield of extract from tanning materials an error is usually made by ignoring the decrease in weight sustained by the material due to loss of soluble matter. This loss, which may run as high as 20%, is only partly accounted for by the formation of sludge during cooling. In a case now being investigated a very considerable loss was sustained, although very little sludge was formed. —D. W.

*Tanning solutions; Filtration of* —. J. G. Parker and J. T. Terrell. J. Soc. Leather Trades' Chem., 1921, 5, 112—115.

EXPERIMENTS are described showing the superiority of the Berkefeld filter candle to filter paper for the determination of soluble matter in tanning extracts. There is loss of soluble matter due to adsorption by the filter paper even when a considerable quantity (200 c.c.) of the filtrate is rejected before evaporating to dryness. Differences of 3.5% and 5.1% of total soluble matter were obtained with mimosa and quebracho extracts respectively. Filter candles are now obtainable, and their use should be made compulsory.—D. W.

*Tannins; Action of* — and the swelling of pelt. G. Grasser. Collegium, 1921, 1—10, 58—67, 105—117. (Cf. J., 1921, 154 A.)

TANNINS have very little repressing action on acid-swollen pelt. Neither sodium chloride nor gallic acid exerts any swelling action on pelt. Formaldehyde has an extraordinarily high repressing action on acid-swollen pelt and neither acids nor alkalis can modify it. Formaldehyde is the most astringent tanning agent known. From their action on swollen pelt, phenol and phenolsulphonic acids should prove good bating materials. Vegetable tannins do not reduce acid-swollen pelt, but the effect is different if the pelt is treated with tannin first and then with acid. Pieces of pelt were treated with different tannin solutions and then with acid and the increase in weight determined and compared with the increase in weight obtained by subjecting untreated pelt to the same acid treatment. From the figures obtained, the initial speed of tannage, C, was calculated. The maximum speed of tannage, J, was similarly obtained by making experiments over longer periods of time, and the intensity of tannage, E, was calculated by dividing the maximum speed of tannage by the time to attain that maximum. Tables are appended giving C, J, and E for a large variety of tanning materials. A comparison of these figures shows that there are tannins which have a comparatively high initial tanning velocity, C, but it is not maintained to the end of the tannage. The synthetic tannins are characteristic in this respect. They have the highest C value and are especially suitable for re-tanning. Tannins which resemble dyes (mangrove, cutch, and logwood) have a very low initial tanning velocity and intensity in spite of their ready solubility. This is due to their feeble tanning properties. Sulphite-cellulose, though a weak tanning agent, has a high tanning velocity. The more favourable behaviour of basic chromic sulphate in comparison with basic ferric sulphate explains why chrome tannage has become of such practical importance. Potash alum gives low values. True tannins do not give high values for C, J and E, but an average for J, 0.45—0.58, C, 0.40—0.49 and E, 0.40—0.54. The values observed for various materials, including tannins and others, are J, 0.36—0.97, C, 0.36—0.93, and E, 0.32—5.62. —D. W.

*Hydrolysis and adsorption [by hide].* V. Kubelka. Collegium, 1921, 135—141.

A CRITICISM of Moeller's work (J., 1920, 730 A). —D. W.

*Synthetic tannins.* C. Immerheiser. Collegium, 1921, 130—132.

A CRITICISM of Moeller's views on synthetic tannins (J., 1921, 154 A). Sulphonation is not the essential principle involved in the preparation of synthetic tannins, but only a means of rendering some of them soluble, and  $\beta$ -naphthalenesulphonic acid, which has none of the characteristics of a tannin, cannot be compared with commercial synthetic

tannins which have a definite tanning action. Ordoval has no harmful action on leather even when used in excess. The synthetic tannins are valuable because of their easy penetrability, and because they dissolve difficultly soluble tannins in mixture with vegetable tanning materials. Ordoval, for dissolving solid quebracho extracts, has replaced sulphiting. In spite of all attacks, the synthetic tannins have gained a firm footing in the leather trade and have attained to great practical importance.—D. W.

*Chrome [tanning] liquors; Acid titration of —.*  
A. W. Thomas and S. B. Foster. J. Amer. Leather Chem. Assoc., 1921, 16, 61—63.

DIAVALE and Das (J., 1920, 758 A) have shown that the maximum increase in acidity is obtained by adding 49 g. or more of sodium chloride to 400 c.c. of solution containing 0.1747 g. of chromium. With samples of chrome liquors diluted to 400 c.c. with distilled water and treated with 50 g. of sodium chloride, the amount of acid found by titrating the boiling solution with  $N/10$  alkali was equal to that determined by the conductance method. The official method for titrating chrome liquors can be made to yield more correct results by adding 50 g. of sodium chloride to the solution before the titration is commenced.—D. W.

*Chrome tanning; One-bath —.* E. Griliches. Collegium, 1921, 127—129. (Cf. J., 1920, 633 A.)

In reply to Stiasny's criticisms (J., 1921, 53 A) the author states that he used feebly pickled pelt for his experiments, equivalent to 2 c.c. of  $N/10$  NaOH per litre, which would be insufficient to influence the figures as Stiasny suggests. It is not easy to show the influence of the acid content of the pelt on the acid taken up from the tanning liquor, and it is probable that Stiasny greatly over-estimates that influence. In Stiasny's suggested method of expressing the basicity of chrome liquors  $Cr:SO_3 = 52.1:X$ , the author considers that X should be termed the acidity.—D. W.

*Aldehyde tannage.* VII. W. Moeller. Collegium, 1921, 117—125.

ALDEHYDE tannage is differentiated from all other kinds of tannage both qualitatively and quantitatively. The peculiar structure of the hide and the energetic tanning action of formaldehyde prevent pelt from absorbing much formaldehyde. Freiberg hide powder has been treated with formaldehyde and all experiments done with it in determining the tanning action of formaldehyde are influenced thereby. Using stronger solutions of formaldehyde and very finely divided untreated hide powder, it is possible to get as much as 27% of formaldehyde taken up by the hide powder, which is almost the same as the amount absorbed by animal charcoal. Pieces of pelt treated with formaldehyde solutions of similar strength do not take up more than 4% of formaldehyde owing to their microscopic structure. The formaldehyde polymers choke the pores of the leather and prevent further penetration.—D. W.

*Iron tannage.* D. D. Jackson and T. P. Hou. J. Amer. Leather Chem. Assoc., 1921, 16, 63—75, 139—159, 202—219, 229—259.

Iron is more basic than either chromium or aluminium. Ferric salts, particularly the sulphate, are very readily hydrolysed, and the ferric hydroxide thus formed seems to pass through the colloidal range so rapidly that it is soon precipitated and this causes hard and brittle leather. Another difficulty with iron tannage is that the ferrous iron in solution, though easily partially oxidised, is completely oxidised only with difficulty. It is necessary to convert the ferrous iron to the ferric state completely and then to keep it in the ferric state

throughout the tanning operation under adverse conditions, and to prevent the separation of any hydrated ferric oxide by maintaining the proper acidity of the liquor employed. Chlorine, sodium nitrate and sulphuric acid, nitric and sulphuric acids, and sodium bichromate are the most suitable oxidising agents, and working details are given for their use in the manufacture of iron tanning liquors. Ferric chloride is more stable towards dilution than ferric sulphate for the same basicity and behaves like chromic chloride and other chromic salts. The oxidation of ferrous sulphate by chlorine is a ready means of securing one-third of acid radical in the form of a chloride.  $Fe(OH)SO_4$  is very unstable whilst more basic ferric chlorides are quite stable. On treating pelt with basic iron liquors, the iron is absorbed by the skin very rapidly. The mineral acid is also taken up by the skin but in less proportion. The tannage is complete after drumming for 1½ hrs. Iron seems to yield a more permanent tannage than alum but the leather will not withstand the action of boiling water. An iron-tanned leather, properly tanned, is not brittle on the grain and does not deteriorate on storing. It is important to rinse the tanned stock, after neutralisation, to remove soluble salts. The harsh feel, due probably to the presence of a large amount of iron oxide in the leather, may be counteracted somewhat by heavy fat-liquoring, oiling, or the use of flour. Not less than 4%  $Fe_2O_3$  should be present in the air-dried leather. Iron-tanned leather compares favourably with other mineral-tanned leathers but the colour is an undesirable feature for some purposes. The basicity ratio of the tanning liquor should vary between one OII equivalent to every 3—5 equivalents of mineral acid radical. The neutralisation should be gradual so that the iron may be uniformly fixed throughout the whole skin. Before dyeing, the leather should be dried out to minimise the chemical reactions between the iron in the skins and the substances employed. A procedure for the chemical analysis of iron-tanned leather is outlined, including the following determinations:—moisture, fat, ash,  $Fe_2O_3$ ,  $Cr_2O_3$ , free mineral acid,  $P_2O_5$ , total  $SO_3$ , hide substance.—D. W.

[*Tannin analysis;*] *Shake method and the filter bell process [of —].* G. Baldracco and S. Camilla. Collegium, 1921, 125—127. (Cf. J., 1920, 496 A.)

A REPLY to Lauffmann's criticism of the authors' modified shake method (Collegium, 1920, 430).—D. W.

*Free sulphuric acid in leather and animal hide; Determination and behaviour of —.* C. Immerheiser. Collegium, 1921, 132—135. (Cf. J., 1920, 731 A.)

MOELLER's criticisms of the determination of free sulphuric acid in leather by the ether-sulphuric acid method (J., 1921, 53 A) are not based on experimental evidence. His claim that leather may contain a considerable amount of chloride is not borne out in practice even with mangrove-tanned leathers, and his assertion that organic matter combines with the sulphuric acid and causes a lower figure to be obtained than the actual amount of acid present is not supported by facts. The amount of sulphuric acid purposely added to a leather can be determined by this method with a fair degree of accuracy. When pelt is brought into contact with a tanning material which has an acid reaction, a process of neutralisation ensues, and this fact can be established experimentally. With increasing tannage the acid figure of the leather diminishes until a constant is obtained which is characteristic for each tannin. The "acid figure" of the finished leather is apparently dependent on the acidity of

the tanning material, so that a leather tanned with a strongly acid tannin will require less acid to neutralise it than a leather tanned with a less acid tannin.—D. W.

Chrome alum solutions. Meunier. See VII.

Sulphonated oils. Hart. See XII.

2,4,6-Trinitroresorcinol. Einbeck and Jablonski. See XX.

#### PATENTS.

Tanning substances; Manufacture of artificial —. M. Melamid. E.P. 137,323, 2.1.20. Conv., 24.12.18.

THE alkali-soluble portion of anthracene oil or "soft pitch" is treated with aromatic sulphochlorides in alkaline solution, and the product is sulphonated.—D. W.

Tanning substances; Process for the manufacture of artificial —. M. Melamid. E.P. (a) 148,268, 23.2.20, and (b) 148,738, 25.2.20. Conv., (a) 19.7.19 and (b) 21.7.19. Addn. to 137,323 (*cf. supra*).

(A) The alkali-soluble portion of anthracene oil boiling between 100° and 300° C. (12 mm.), or of soft pitch (100 kg.), is sulphonated at 30°–40° C. with an equal weight of 100% sulphuric acid until the mass is soluble in water, when the product is dissolved in water, neutralised with sodium hydroxide, and the bulk of the sodium sulphate removed by evaporation and crystallisation. A solution of 28 kg. of sodium hydroxide is added to the product, and the mixture is agitated with a solution of 100–110 kg. of *o*- and/or *p*-toluenesulphonyl chloride in 200 l. of benzene at normal temperature until the whole of the sulphonyl chloride has entered into reaction. The product is used for tanning without further treatment. (B) The product as obtained above, but omitting the treatment with sulphonyl chlorides, is used direct for tanning.—L. A. C.

Substances precipitating glue [tanning agents]; Manufacture of —. A. Römer, Assr. to A. Kuttroff. U.S.P. 1,375,976, 26.4.21. Appl., 9.5.17. Renewed 29.6.20.

A NAPHTHALENESULPHONE is sulphonated and the product is treated with formaldehyde, yielding a substance capable of precipitating glue from its solutions.—L. A. C.

Leather; Process for impregnating and loading —. H. Burger. G.P. 303,204, 15.5.14.

THE addition of naphthalene, alone or in combination with substances such as fat, paraffin, tallow, wax, resin, asphalt, or caoutchouc, to impregnating or filling agents for leather, improves their penetrating power. Sulphur may be utilised as an impregnating agent by dissolving it in naphthalene. The dried, heated leather is steeped in the solution at 100° C. for  $\frac{1}{2}$ –1 min. The product may be readily dyed.—W. J. W.

Hides; Depilation of —. W. Rautenstrauch. G.P. 304,251, 8.4.17.

BARIUM hydroxide solution, of a concentration above N/10, forms a satisfactory depilatory agent for hides. The waste solution may be used again after addition of more barium hydroxide, and its efficiency is not affected by presence of albuminoid matter from the hides. After treatment the hides may be freed from barium hydroxide by washing with water containing a small amount of sulphuric, hydrochloric, lactic, or butyric acid, or of an acid salt.—W. J. W.

Hides; Process for unhairing — by the action of enzymes. R. Hilgermann and M. Emmerich. G.P. 334,526, 26.10.18.

CULTURES of *B. emycoides tumescens*, *megatherium*, and *mesentericus* are prepared using a mixture of beef, horse-flesh, eggs, and lactose, and of *Mucor* and *Aspergillus* using yeast, glucose, resin, and asparagin, whilst mixed cultures are prepared by means of glycerin and peptone. By suitable treatment enzyme preparations are obtained from these bacteria and moulds in 8–12 weeks. The hides, which have been previously softened with acid and alkali, are placed in water and treated with the enzyme preparation, in the proportion of 300–500 c.c. per 100 kg. of hide, for 24–96 hrs. at 22°–25° C. —W. J. W.

Substances precipitating glue [tanning agents]; Manufacture of —. A. Römer, Assr. to A. Kuttroff. U.S.P. 1,375,975, 26.4.21. Appl., 23.11.14. Renewed 2.4.20.

SEE G.P. 293,042 of 1913; J., 1916, 1072.

Casein specially adapted for the production of imitation horn; Manufacture of —. A. Bartels. E.P. 162,301, 16.1.17.

SEE U.S.P. 1,320,666 of 1919; J., 1919, 955 A.

## XVI.—SOILS; FERTILISERS.

Soil organic matter and organic carbon; Dry combustion method for the simultaneous determination of —. J. W. Read. J. Ind. Eng. Chem., 1921, 13, 305–307.

A WEIGHED portion of the soil is digested with dilute hydrochloric acid and hydrofluoric acid to remove carbonates and hydrated minerals, and the insoluble portion is collected on a layer of asbestos in a combustion boat having a perforated bottom (*cf.* Rather, J., 1918, 478 A). After drying, the boat is transferred to a silica combustion tube which is heated by a three-unit electric furnace; the tube is packed as follows:—oxidised copper gauze, combustion boat, asbestos and pumice coated with cerium dioxide, copper oxide, asbestos and pumice coated with cerium dioxide, and copper gauze filled with lead peroxide. The latter layer is maintained at 300°–320° C. The carbon-dioxide is absorbed in bulbs filled with soda-lime.—W. P. S.

Humic acids. S. Odén. Kolloid-Chem. Beih., 1921, 11, 75–260.

A COMPREHENSIVE review of the humic acids. These acids are subdivided into humic acid, hymatomelanic acid and fulvic acid. Humic acid with ammonia yields salts and adsorption compounds. Humic acid has an equivalent of 340±10; it is tetrabasic, insoluble in water, but readily forms colloidal solutions. It has the composition  $C_{10}H_{10}O_4(CO_2H)_4$ , and the anion is almost black in colour. Hymatomelanic acid is yellowish-brown and has an equivalent weight of 200; its salts are insoluble in alcohol, but easily form highly dispersed suspensions. Humus colloids exert a protecting action on clays which is of the same order as that of other protective colloids, but is specific to certain varieties of clay and electrolytes. Limed samples of humoid soil bind more water than unlimed samples, but the difference in amount is not of practical importance. The limed humus gives up its water more readily than the unlimed humus. After liming the water appears to be a little more firmly held and consequently is less easily taken up by plants.—J. F. S.

**Boron in relation to the fertiliser industry.**  
J. E. Breckenridge. J. Ind. Eng. Chem., 1921,  
13, 324—325.

DURING recent years potash has been obtained from many new sources and some of the raw materials used have contained boron; cases of injury to crops have been noticed where such fertilisers have been used. Maize and beans are injured by the application of 6 lb. of borax per acre; the growth of potatoes appears to be stimulated with 4—6 lb. of borax per acre, but with 10 lb. there is decrease in root formation. With plenty of moisture the plants recover gradually, but would probably die in a dry season.—W. P. S.

**Sulphur; Non-biological oxidation of elementary — in quartz media.** W. H. MacIntire, F. J. Gray, and W. M. Shaw. J. Ind. Eng. Chem., 1921, 13, 310—313.

SULPHUR is oxidised by moist quartz under sterile and non-sterile aerobic and anaerobic conditions. The oxidation is accelerated by the presence of limonite and is decreased by that of iron or magnesium carbonate.—W. P. S.

**Cellulosic residue of leaves.** A. J. J. Vandevclde. Bull. Soc. Chim. Belg., 1920, 29, 317—322.

THE insoluble cellulose residue (residue remaining after an acid and alkali treatment of the dried and powdered substance) of various leaves, including the vine, lilac, gooseberry, raspberry, and lime, at various periods of growth, was determined. Very irregular variations were found at different periods, and in different years, for the same kind of leaf, and no general relationships were apparent similar to those previously found for the ash, water or starch content at different stages of development of the leaf. The acid residue and the alkaline residue of the dried leaf showed analogous variations.  
—G. F. M.

**Cryptogamic diseases of plants. Anticryptogamic product.** Lance. See XIX b.

**Humic acids.** Eller. See XX.

#### PATENTS.

**Fertilisers; Manufacture of —.** Badische Anilin u. Soda Fabrik. E.P. (A) 145,036 and (S) 145,037, 14.6.20. Conv., 29.7.16 and 26.3.17.

(A) A NON-DELIQUESCENT and non-explosive fertiliser is obtained by mixing ammonium nitrate and potassium chloride in the presence of a small quantity of water, or by mixing ammonium nitrate, containing a small percentage of moisture, with dry potassium chloride. Crude potassium chloride containing about 64% KCl may be used. (B) Ammonium nitrate in the dry state is mixed with potassium chloride or potassium sulphate in absence of water.—W. J. W.

**Fertiliser, and process for making same.** E. H. Sams. E.P. 161,777, 17.2.20.

UNDERGROUND chalk is mixed with house refuse which has been pulverised and screened, or with the ashes of the same, and the mixture is then milled. The addition of the house refuse facilitates the grinding of the chalk owing to absorption of its moisture.  
—W. J. W.

**Fertiliser; Process of producing —.** M. Shoeld, Assr. to Armour Fertilizer Works. U.S.P. 1,375,115, 19.4.21. Appl., 13.9.20.

A SUITABLE material is added to wet precipitated phosphate to prevent the latter becoming dusty after drying.—W. J. W.

**Lime; Production of catalytic — for fertilising.**  
R. Mandelbaum. G.P. 333,484, 22.12.18.

THE fertilising properties of lime are improved by the addition of small amounts of calcium sulphate, sulphite, or sulphide. By the action of water on calcium sulphate and sulphide in the soil, heat is generated.—W. J. W.

#### XVII.—SUGARS; STARCHES; GUMS.

**Sucrose; Decomposition of — during milling.**  
J. N. S. Williams. Int. Sugar J., 1921, 23, 271—272.

IT is believed that an appreciable loss of sucrose must occur during the operation of milling under present-day conditions, that is, when the cane is finely shredded, and when the filter-press washwaters and the thin juices from the last unit are used for maceration. It was observed in the case of Plantation A, applying a moderate crushing and maceration, that the extraction was 89.1%, the fibre content, 13.3, the polarisation of the first mill juice, 16.6, the purity of the first mill juice, 88.3, and the calculated sugar in the cane, 279.4 lb. per ton; while on Plantation B, in which the cane was subjected to the extreme degree of crushing and compound maceration, the extraction was 96.34, the fibre content, 13.76, the polarisation of the first mill juice, 17.5, the purity of the first mill juice, 87.55, and the calculated sugar in the cane, 268.2 lb. per ton. Thus, Plantation A dealing with an inferior cane showed 279.4 lb. of sugar per ton of cane compared with 268.2 lb. in the case of Plantation B with a much better cane.—J. P. O.

**Sucrose; Neutral polarisation modification of the Clerget method of determining —.** C. A. Browne. Int. Sugar J., 1921, 23, 276—281.

REPLYING to Jackson and Gillis (J., 1921, 361A), the author agrees that in the case of Method 2 prescribed in Bull. 375 (J., 1920, 634A) the errors in determining the positive and negative constituents do to a considerable extent neutralise each other, though his experiments show that compensation is perfect only at a concentration of about 13 g. of sucrose per 100 c.c. However, he points out that in the case of Method 3 (loc. cit.) for the analysis of beet products, the errors remain uncompensated, since the ammonium chloride is present only when making the negative reading, none being added for the positive reading as in Method 2. In Method 3, therefore, the final error may be considerable.  
—J. P. O.

**Sucrose; Determination of — by the inversion method.** V. Sázkavsky. Z. Zuckerind. Czechoslov., 1921, 45, 227—229, 235—238.

IS completely precipitated by basic lead acetate and for the clarification of solutions of raw (beet) sugar products the author recommends tannin solution followed by basic lead acetate solution. Although commercial tannin is optically active it gives rise to no error. A solution containing the normal weight (26 g.) of molasses may be treated with 10—15 c.c. of a 6% tannin solution and then, after shaking round, with 10—12 c.c. of basic lead acetate solution; for twice the normal weight of raw sugar it is usually sufficient to employ 10—20 drops of the tannin solution, followed by 1—2 c.c. of basic lead acetate. Solutions thus treated require no further clarification after inversion. Determinations of the inversion constant under Clerget-Herzfeld conditions, for different concentrations of pure sucrose, practically confirmed Herzfeld's formula. The presence of 1 g. of molasses ash in the solution raised the constant by 0.2; and in inverting 65 g. of molasses it is therefore better to

employ the factor 132.66 than the lower one corresponding to 65% solution of pure sucrose. For the inversion of molasses 5 c.c. of acid is not always sufficient, and it is advisable to use 6.5 c.c. Stanek's method for eliminating the error due to optically active non-sugars (J., 1914, 705) is considered more convenient than those proposed by Saillard and Andriik and Stanek (cf. J., 1911, 63).—J. H. L.

*Dry substance of sugar products; Determination of — by means of the refractometer in the presence of crystals.* H. Kalshoven. *Archief Suikerind. Nederl.-Indië*, 1920, 28, 913—917. *Int. Sugar J.*, 1921, 23, 289—290.

In determining the dry substance of sugar products containing crystals, the author no longer uses the lower prism of the refractometer, the massecuite or molasses being pressed with the aid of a small glass plate (reinforced with a piece of copper sheet) against the upper prism, and the reading taken at least 10 mins. after turning on the cooling water.—J. P. O.

*Sugars; Configuration of some —.* [Use of invert sugar in titrating boric acid.] J. Boëseken and H. Couvert. *Rec. Trav. Chim.*, 1921, 40, 354—380.

THE electrical conductivity and optical rotation, with and without the addition of boric acid, of a number of substances of the sugar group have been determined. Invert sugar, prepared by heating sucrose for 1 hr. at 100° C., with 0.25 times its weight of 0.1% aqueous hydrochloric acid and neutralising the product, may with advantage be employed in titrating boric acid with barium hydroxide. Very accurate results are obtained in N/10 solution with phenolphthalein as indicator if sufficient invert sugar be used to provide 5 mols. of levulose for every mol. of boric acid. (Cf. J.C.S., July).—C. K. I.

*Starch; Studies on —.* A. Reyher. *Bull. Soc. Chim. Belg.*, 1920, 29, 309—317.

THE gelatinisation of starch can be conveniently followed microscopically at ordinary temperatures by using a solution containing about 0.135 mol. of alkali hydroxide per litre. A similar action is produced by 4N or 5N acids and by certain concentrated salt solutions, such as 30% ammonium nitrate, 15% potassium thiocyanate, etc. The reverse action is produced if to 100 c.c. of a starch paste, 80 g. of sodium sulphate, 40 g. of ammonium or magnesium sulphate, etc., is added, a complete coagulation resulting in a few minutes regardless of the temperature. Under ordinary conditions this retrogradation takes 10 or 12 days, and is then only partial. The microscopical observations may be interpreted by postulating that the starch mucilage is a suspension of swollen and partially burst granules consisting of an internal substance and an envelope which is impenetrable by water unless at an elevated temperature or in presence of an alkali, etc., when it becomes hydrated and allows the internal material to form a voluminous gel or sol. The coagulum produced by sodium sulphate etc., is composed of amorphous granules, smaller in size than the swollen granules of the mucilage, and formed by the shrinking of the envelope and the consequent expulsion and coagulation of the internal sol. Starch solutions formed by heating the mucilage at 120°—150° C. exhibit a similar phenomenon of coagulation, and the precipitate formed may readily be isolated by taking advantage of its resistance to diastase. On drying it forms a hard, warty mass, resistant to all reagents except potash, but still showing under the microscope its constituent globules. It is dissolved by water at 150° C., and the solutions resemble those of the original starch, but are characterised

by giving a deeper blue colour with iodine, and by yielding an almost theoretical quantity of maltose when treated with malt extract. It appears, therefore, to be composed exclusively of a polysaccharide (amylose), whilst the original starch contains in addition a more refractory substance originally designated by Maquenne amylopectin (cf. J., 1905, 630). Amylose may therefore be characterised as a solid, relatively soluble, in the starch granules; colloiddally dissolved and coagulable in the internal fluid of the granules in the mucilage, in starch solutions, and in solutions of the coagulum; solid and attackable with difficulty in the coagulum. Amylopectin is met with as a solid, dense and resistant in the envelope of the starch granules; as solid, but swollen permeable by water and liquefiable by malt extract, in the granular envelopes of the mucilage; and also dissolved in the starch solutions.—G. F. M.

*Cellulose dextrins.* Samec and Matula. See V.

#### PATENTS.

*Sugar cane mills.* F. J. de Bruin. E.P. 162,583, 20.4.20.

*Separating floating impurities.* E.P. 162,200. See I.

*Decolorising carbon.* E.P. 139,156. See IIs.

*Decolorising charcoal.* E.P. 162,117. See IIs.

*Oxalic acid.* G.P. 329,591. See XX.

### XVIII.—FERMENTATION INDUSTRIES.

*Extract yield from raw grain (maize and rice); Determination of —.* W. Windisch and P. Kolbach. *Woch. Brau.*, 1921, 38, 57—59, 63—64, 76—77.

A COMPARATIVE study of various methods. In the one recommended 40 g. of the maize or rice is mixed with 150 c.c. of water and 100 c.c. of a filtered malt extract (made by digesting 1 pt. of malt with 4 pts. of water for 1 hr. at 30° C. with frequent stirring). Next morning the mash is heated rapidly to 70° C., then in 40 mins. to 80°, and then boiled for 5 mins. on a sand-bath. After cooling to 50° it is treated with a further 100 c.c. of malt extract and the temperature is maintained at 50° for  $\frac{1}{2}$  hr., raised to 70° in 20 mins., maintained at 70° for 25 mins., raised to 75° in 5 mins., and maintained at 75° for 30 mins. The cooled mash is made up to 450 g. before filtering. The method of calculation is explained fully.—J. H. L.

*Malt analysis; Comparative experiments respecting the most convenient method of calculation and the accuracy attainable in the pyknometric determination of extract in —.* H. Keil. *Woch. Brau.*, 1921, 38, 95—96.

IN accurate determinations of wort gravities by the pyknometer, tedious corrections for air displacement under different conditions of barometric pressure may be avoided by the use of a "compensation pyknometer" (i.e., a counterpoise such as a pyknometer suitably loaded with shot and hermetically sealed, having approximately the same weight and volume as the pyknometers in use when the latter are filled with water). The exact weight of the counterpoise is determined once for all, and the extra weights required in one pan or the other to balance a pyknometer filled with water or wort against the counterpoise are added to or subtracted from the weight of the latter. This method has been adopted in the analytical laboratory of the Versuchs und Lehranstalt in Berlin.—J. H. L.

*Enzymes; Actions between* — H. Van Laer. Bull. Soc. Chim. Belg., 1919, 28, 340—346.

THE maceration juice, separated at the moment of autolysis from a mixture of dried yeast and water, contains an invertase, which is sensitive to both active and passive papain and to amylase. This sensitiveness is not shown by the juice separated after autolysis or by the limpid extracts from yeast killed by acetone. The liquids obtained by macerating yeast killed by acetone with active solutions of papain or amylase show a notable increase in inverting power, which is apparently due to the difference between the increase resulting from the liberation of invertase masked by the protoplasmic substances, and the diminution caused by a greater sensitiveness of the freshly liberated enzyme to the products of digestion of its cellular substrate.

—W. G.

*Fruit wine; Preparation of* — from frozen or frost-bitten fruit, with special reference to changes in the juice corresponding to the severity of freezing. A. Widmer. Schweiz. Apoth.-Zeit., 1920, 58, 339—344. Chem. Zentr., 1921, 92, II., 878—879.

EXPRESSED juice from frost-bitten fruit (apples and pears) has a low sp. gr. owing to its relatively large amount. Mere freezing of the fruit produces no perceptible change in the constituents of the juice, and does not preclude vinification provided the fruit is thawed without delay, at the lowest possible temperature, and the expressed juice is fermented at once. In frost-bitten fruit, on the other hand, there occurs a decrease in the acidity and tannin-content of the juice, part of the tannin being precipitated in combination with proteins; such fruit yields mild but unstable juices which sometimes have an unpleasant flavour and odour. Frost-bitten unripe fruit is only fit for vinification if free from the last-mentioned defects, and in any case it is advisable to employ pure yeast for the fermentation. All fruit wine made from frozen fruit should be consumed as soon as possible.—J. H. L.

*Phytase.* Collatz and Bailey. See XIXA.

*Tartaric acid etc.* Voss. See XX.

#### PATENTS.

*Alcoholic fermentation; Process for accelerating* — Aktiebolaget Astra, Apotekarnes kemiska fabriker. G.P. 334,866, 7.5.14. Conv., 6.5.13.

THE liquid to be fermented is treated with one or more salts of organic acids, other than ammonium salts, or with neutralised solutions containing organic acids produced biochemically. Alkali salts of acids of the formic and glycolic acid series are particularly active, producing an acceleration of 30—100%, with increased attenuation in many cases.—J. H. L.

*Fat in microbes.* G.P. 332,411. See XII.

*Extracting ferments etc.* G.P. 334,660. See XX.

#### XIXA.—FOODS.

*Milk; Detection of the addition of sodium carbonate to* — A. J. J. Vandeveld. Bull. Soc. Chim. Belg., 1920, 29, 52—54.

THE author relies on an ash analysis. The ash of milk normally contains Na 6.0%, K 20.4%, P<sub>2</sub>O<sub>5</sub> 26.3%. The addition of sodium carbonate will alter the ratio of Na+K to P<sub>2</sub>O<sub>5</sub>, as well as the ratio Na to K.—W. G.

*Goat's milk; Detection of* — in cow's milk. W. Austen. Milchw. Zentr., 1921, 50, 125—127.

THE method depends on the insolubility of the casein of goat's milk in 25% ammonia, in which the casein of cow's milk is completely soluble. It is only applicable to fresh milk. Potassium bichromate should be used as a preservative, as formaldehyde affects the solubility of the casein. The cream should first be separated as completely as possible by centrifuging in a Gerber tube stoppered at both ends, and removing by means of a fine pipette. About 20 c.c. of the separated milk is thoroughly mixed with 2 c.c. of 25% ammonia in the Gerber tube and beaten in a water bath for about  $\frac{1}{4}$  hr. The tube is then placed in a centrifuge with the graduated part outwards and rotated at about 1200 revs. for some minutes. Cow's milk gives little or no precipitate, whereas that from pure goat's milk about half fills the narrow graduated tube. Less than 20% of goat's milk in cow's milk can, however, not be detected with certainty. The method can be applied to approximate quantitative estimations.

—H. C. R.

*Lard; Boehmer's method for detecting tallow in* — Vitoux and C. F. Muttelet. Ann. Falsif., 1921, 14, 86—92.

FURTHER experiments with the method previously described (J., 1921, 126 A) confirm its usefulness. As little as 5% of beef or mutton fat can be detected in lard, the value (2G—A) falling below 68; even in the case of lard adulterated with beef or mutton fat together with fats rich in liquid glycerides, e.g., horse fat, the method loses none of its sensitiveness. It is equally effective in detecting hydrogenated fish oils. Fats poor in solid glycerides, however, such as oleomargarines and horse tallow, escape detection unless present to the extent of more than 20—30%, but it appears possible that a modification of the method may overcome this difficulty.—J. H. L.

*Phytase; Activity of* — as determined by the specific conductivity of phytin-phytase solutions. F. A. Collatz and C. H. Bailey. J. Ind. Eng. Chem., 1921, 13, 317—318.

CHANGES in the specific conductivity of an aqueous solution of phytin and phytase indicate the progress of the hydrolysis of the phytin. In the case of phytase prepared from wheat bran, the most favourable temperature for complete hydrolysis appears to be about 55° C., although the hydrolysis proceeds more rapidly at 60° C. during the first 15 mins.

—W. P. S.

*Wheat flour grades. Electrical conductivity of water extract.* C. H. Bailey and F. A. Collatz. J. Ind. Eng. Chem., 1921, 13, 319—321.

THE specific conductivity of water extracts of wheat flour varies with the time and temperature of extraction; the highest values are obtained at about 60° C., and the conductivity is due chiefly to inorganic phosphates resulting from the hydrolysis of phytin by phytase. To compare different flours it is necessary to use a uniform procedure in the preparation of the extracts, and it is suggested that 10 g. of flour be heated at 25° C. for 30 mins. with 100 c.c. of conductivity water, the mixture then centrifuged, and the conductivity of the clear extract determined at 30° C. with a dip electrode. The specific conductivity of flour extracts runs parallel with the ash content and can be used as an index of the grade of the flour.—W. P. S.

#### PATENTS.

*Cooked food products from wheat grain; Manufacture of* — and apparatus therefor. C. V. Rowell. E.P. 140,457, 18.3.20. Conv., 18.3.19.

WHOLE wheat grains are heated at about 460°—



500° C. for about  $\frac{1}{2}$  min. in presence of highly superheated steam under a pressure of about 1000 lb. per sq. in., and the product is ejected from the horizontal, cylindrical heating chamber by a sudden release of the pressure.—H. H.

*Blood-meal; Manufacture of* — G. A. Krause. G.P. 332,434, 19.8.17.

Blood which has coagulated without heating, and in which the clots have been subsequently broken up into fine particles, is atomised and dried by a current of hot air, yielding a meal completely soluble in water with the exception of the fibrin.

—L. A. C.

*Plants; Process for improving the flavour and odour of food- or fodder-yielding — of the Cruciferae or related families.* H. Brün. G.P. 334,274, 18.10.17.

THE comminuted and partially or completely dried material is moistened with a very dilute solution of hydrogen peroxide, then tightly compressed for 48 hrs., and afterwards spread out and dried. Products thus obtained from beets and the like may be used as diluents or substitutes for flour or soup powders or may be employed in the manufacture of jam.—J. H. L.

*Pickling brine; Process for recovering proteins contained in* — G. Coblitz. G.P. 334,322, 10.12.16.

THE proteins, separated from solution by fractional thermal coagulation, are cooled at once by means of water or otherwise, and the different fractions are subsequently mixed and dried as usual.—J. H. L.

*Lupin seeds, horse chestnuts and the like; Removal of bitter substances from* — P. Schmidt. G.P. 334,589, 5.12.18.

THE seeds, freed from shells, are treated for 1 hr. at 95°–100° C. with a dilute solution (1.25) of the waste mother liquor from potash manufacture, and then twice more with similar solution, for 2 hrs., each time at 50° C., with continuous stirring, and finally the seeds are well washed with water, dried, and ground if necessary.—J. H. L.

*Fodder from straw (e.g., from cereals or leguminous plants); Preparation of a — by digestion of the comminuted material [with alkali solution].* Veredelungsges. für Nahrungs- und Futtermittel m.b.H. G.P. 335,155, 27.3.18. Addn. to 305,641 (J., 1919, 789 A).

THE alkaline digestion liquors are used over and over again until they become acid, and each charge of straw is digested in stages with successive portions of liquor, each fresher than the preceding one. The alkali is thus completely utilised and digestion is accelerated.—J. H. L.

*Cooling chocolate and other materials; Machine for* — E. Savy. U.S.P. 1,375,734, 26.4.21. Appl., 1.3.19.

SEE E.P. 124,196 of 1919; J., 1920, 78 A.

## XIXB.—WATER PURIFICATION; SANITATION.

*Boiler feed water; Purification of* — F. Blumen-thal. Z. angew. Chem., 1921, 34, 189–192.

A REVIEW of the lime-soda, baryta, Permutite, and Neckar processes for softening boiler feed water. The lime-soda process can only reduce the hardness to a limit of 2 or 3 degrees on account of the solubility of calcium and magnesium carbonates, and in addition soluble sodium salts and about 10–15 g. of sodium hydroxide per cub. m. are left in the water and pass into the boilers, which must there-

fore be blown out periodically. In the baryta and Permutite processes, on the other hand, smaller amounts of soluble salts are left in the feed water. In the Neckar process, heating the feed water to 80° C. cannot cause the precipitation of the calcium and magnesium carbonates; such precipitation must be due solely to the soda alkalinity of the boiler water which is returned to the purifier. Moreover, the statement that in this process the carbonic acid is entirely precipitated is quite incorrect, since it is all converted in the purifier into sodium bicarbonate, is fed into the boiler, and is there expelled with the steam, the residual normal carbonate being returned to the purifier and the above operation repeated. Magnesium salts are only completely precipitated when sufficient sodium hydroxide is formed in the boiler by hydrolysis of the carbonate unless additional hydroxide is supplied to the purifier, which materially increases the cost of the process.—G. F. M.

*Cryptogamic diseases of plants; Use of coloured screens for combating* — R. Lance. Comptes rend., 1921, 172, 1201.

THE spray is prepared by mixing 650 g. of ultramarine blue, 250 g. of ultramarine green, and 100 g. of ultramarine violet with several litres of water, adding 2 kg. of calcium sulphate and 1350–1400 g. of quicklime, and when the colouring is uniform, stirring in 1 kg. of aluminium sulphate or 15 kg. of alum. The precipitated alumina fixes the dye and the whole mixture is diluted to 400 litres and is ready for use. The deposit formed on the plants by the use of this spray acts as a colour screen, permitting only blue, violet, and ultra-violet rays to pass.—W. G.

*Anticryptogamic product.* R. Lance. Comptes rend., 1921, 172, 1201–1202.

THE material recommended is prepared by dissolving 1 kg. of zinc sulphate in 100 l. of water, adding 500 g. of lime to precipitate the zinc as hydroxide, and then 80–100 g. of an ultramarine colour. Finally 250 g. of aluminium sulphate is added and the whole is well shaken and after filtering is ready for use. This mixture, it is claimed, combines the antiseptic action of zinc with the microbicidal action of the blue, violet, and ultra-violet rays.

—W. G.

## PATENT.

*Water; Process and apparatus for removing dissolved oxygen from* — L. and C. Steinmüller. G.P. 334,181, 28.3.17.

THE water is passed slowly over an extensive surface of iron in a direction substantially parallel to the surface. Vessels or drums packed with a series of concentric sheet iron cylinders may be used. The process is more effective than the use of filters containing iron shavings, and the apparatus lasts longer and is more easily cleaned than the latter.

—J. H. L.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Morphine; Chemico-toxicological detection of* — D. Ganassini. Boll. Chim. Farm., 1921, 60, 2–5.

IN the test for morphine given by Denigès (J., 1911, 47), the copper sulphate and hydrogen peroxide may be replaced by potassium ferricyanide and ammonia; the reaction is then applicable to the detection of morphine in blood and in non-putrefied animal organs, but is not very sensitive. The copper sulphate may be replaced also by hæmatin, which readily catalyses hydrogen peroxide, the

hamatin of human blood being far more active in this respect than that of the blood of animals. For the detection of morphine in blood, Denigès' test must be modified as follows: 0.25 c.c. of blood is diluted to 10 c.c. with water and the solution treated with nitric acid (about 10 drops) until a brown precipitate of globin and hamatin is formed. The liquid is filtered and the colourless or almost colourless filtrate treated with 1 c.c. of ammoniacal cuprammonium sulphate solution, prepared by adding 10 drops of hydrogen peroxide solution (1 vol. perhydrol to 1 water) to a mixture of 20 c.c. of concentrated ammonia with 5 c.c. of 0.5% copper sulphate solution. With blood free from morphine, the liquid remains colourless, whereas the presence of a minute proportion of morphine rapidly produces a more or less intense pink coloration. This procedure serves also for the detection of morphine in milk or urine, addition of nitric acid being then unnecessary; in these cases, however, the reaction is less sensitive than with blood. The above reaction is given neither by hydroxydimorphine, nor by the alkaloids accompanying morphine such as dionine and peronine, but it is shown, contrary to the statement of Denigès, by heroine. The latter gives the following specific reactions, which serve to distinguish it from morphine: When heated with sulphuric acid it yields acetic acid, or in presence of alcohol, ethyl acetate; when treated with concentrated nitric acid it becomes successively yellow, greenish-blue, and yellow slowly in the cold, but rapidly when heated; when treated with 2 c.c. of a solution of urotropine (hexamethylenetetramine) in concentrated sulphuric acid it turns first golden-yellow, then orange, and finally dark blue.—T. H. P.

*Iodotannic syrups; Preparation of —. Rapid determination of combined iodine.* T. Ugarte. Anal. Asoc. Quím. Argentina, 1921, 9, 5—16.

METHODS are described for avoiding loss of iodine in the preparation of iodotannic syrups. Two rapid methods are described for the estimation of combined iodine in two different types of preparation. The second method, which is applicable in both cases, consists in titrating the syrup with N/10 mercuric chloride until a permanent opalescence is obtained; each molecule of mercuric chloride corresponds to three atoms of combined iodine.

—G. W. R.

*Humic acids; Synthetic and natural —.* W. Eller. Brennstoff-Chem., 1921, 2, 129—133.

Humic acids have been obtained by the oxidation of phenols in alkaline solution (J., 1920, 717A). The chemical reactions of the products resemble those of the natural humic acids. (Cf. J.C.S., July.)—W. P.

*2,4,6-Trinitroresorcinol (styphnic acid).* H. Einbeck and L. Jablonski. Ber., 1921, 54, 1084—1089.

STYPHNIC acid, m.p. varying from 174° C. to 179°—180° C., is obtained by the oxidation of quebracho extract with nitric acid (sp. gr. 1.4). For its characterisation the mono- and di-potassium salts have been prepared by the regulated addition of alcoholic potassium acetate solution to styphnic acid dissolved in alcohol, and the mono- and di-sodium salts by the use of alcoholic sodium hydroxide. All the salts, the mono-sodium compound in particular, explode violently when heated. (Cf. J.C.S., July.)—H. W.

*Tartaric acid and acid potassium tartrate; Manufacture of —.* H. Voss. Chem.-Zeit., 1921, 45, 309—313, 335—337, 360—364, 411—412.

THE manufacture of acid potassium tartrate, with details of the necessary plant is described. An

extensive survey of the manufacture of tartaric acid by both the neutral and the acid process has been made, for details of which the original paper should be consulted.—W. J. W.

*ββ-Dichloroethyl sulphide.* Delépine, Fleury, and Ville. Comptes rend., 1921, 172, 1238—1240.

ββ-DICHLOROETHYL sulphide when prepared from ethylene and sulphur chloride is never pure. It contains substances which are apparently isomeric with it, and on distillation under reduced pressure yields a small amount of hydrogen chloride. It cannot be used satisfactorily as a solvent for cryoscopic measurements.—W. G.

*Cyanic acid and urea; Synthesis of — by oxidation of ketones, acids, and amines in presence of ammonia.* R. Fosse and G. Laude. Comptes rend., 1921, 172, 1240—1242.

CYANIC acid and subsequently urea, by tautomerisation, have been obtained by the oxidation in the presence of ammonia of ketones, mono- and dihasic acids, phenols, alcohols, and amines. The yield of urea varies considerably with the substance used, and in the case of amines, those capable of giving formaldehyde by oxidation give the highest yield.—W. G.

*Chloroform; Manufacture of — from ethyl alcohol, and the mechanism of the reaction.* S. Ochi. Kogyo-Kwagaku Zasshi (J. Chem. Ind. Japan), 1921, 24, 209—228.

CHLOROFORM was prepared by passing chlorine into a mixture of ethyl alcohol and milk of lime at 60° C., the concentration of alcohol being 10%, and the quantity of calcium hydroxide ca. 300 g. per litre of alcohol, and that of chlorine 0.75—0.87 mol. per mol. of calcium hydroxide; 63.5—65.5% of the chlorine and 54.4—54.9% of the alcohol were utilised. Magnesium oxide, barium oxide, sodium and potassium hydroxides gave poorer yields than calcium oxide, and ferric hydroxide had no catalytic action on the process. The products of the reaction were examined quantitatively, and it is concluded that reactions corresponding to the following equations take place:  $2C_2H_5OH + 8CaOCl_2 = 2CHCl_3 + 2Ca(OH)_2 + 5CaCl_2 + Ca(HCO_3)_2 + 2H_2O$ ;  $2C_2H_5OH + 10CaOCl_2 = 2CHCl_3 + 2Ca(OH)_2 + 7CaOCl_2 + CaCO_3 + CO_2 + 3H_2O$ ;  $C_2H_5OH + 2CaOCl_2 = CH_3COOH + 2CaCl_2 + H_2O$ . 65—75% of the formic acid produced is decomposed into carbon dioxide and water by further oxidation and one-fourth of the acid remains unchanged in the residue. About two-thirds of the alcohol used is utilised in the formation of chloroform and the greater part of the remainder is converted into acetic acid. Carbon dioxide is not produced directly from the alcohol. The explosive reaction in manufacturing chloroform is due to violent evolution of oxygen by the decomposition of calcium hypochlorite at the high temperature produced during the oxidation of the alcohol. It can be avoided by operating at a lower temperature.—K. K.

*Ethyl chloride; Rate of evaporation of — from oils.* C. Baskerville and M. Hirsh. J. Ind. Eng. Chem., 1921, 13, 322—323.

THE rate of evaporation of ethyl chloride from oils (maize oil) quickly acquires a definite and fairly constant velocity when the mixture contains 13—14% by vol. of ethyl chloride. If a mixture originally containing 15% is used, the uniform speed of evaporation is established within 10 mins. for surface exposures.—W. P. S.

*Acetic acid and anhydride.* L. Crismer and J. Timmermans. Bull. Soc. Chim. Belg., 1920, 29, 46—52.

THE rule of mixtures for critical temperatures of

solution does not apply to mixtures of acetic acid and its anhydride. By means of determinations of critical temperatures of solution with a Russian petroleum it is shown that phosphoric anhydride is a suitable reagent for dehydrating acetic acid.

—W. G.

*Catalysts; Influencing of the activity of —. III.*

*Oxidative catalytic dehydrogenation of alcohols.*

I. K. W. Rosenmund and F. Zetzsche. Ber., 1921, 54, 1092—1098. (Cf. J., 1921, 321 A, 368 A.)

THE catalytic oxidation of a primary alcohol may lead to the production of aldehyde, acid, ether, and condensation products. The reaction has been studied in the case of benzyl alcohol under conditions which do not involve vaporisation and with the object of limiting the scope of the change to the production of benzaldehyde. Benzyl alcohol only gives about a 3% yield of the latter when oxidised by oxygen in boiling cumene solution and this yield is not very greatly increased in the presence of copper, silver, magnesium, platinum, nickel, aluminium, zinc, antimony, or carbon. Amongst these almost equivalent catalysts, a selective action can be induced in the cases of copper, nickel, and silver by the suitable addition of quinoline, the production of benzaldehyde being favoured in the case of copper and the formation of ether and condensation products in the cases of nickel and silver, thus giving an example of the transformation of slightly active catalysts into powerful and specific substances. The dehydrogenating action of catalytically excited oxygen and of nitro-groups is additive, the latter being introduced preferably in the form of *m*-dinitrobenzene. The best yields of aldehydes from alcohols are obtained by the oxidation of an equimolecular mixture of alcohol, quinoline, and *m*-dinitrobenzene in cumene solution in the presence of copper.—H. W.

*Alcohols; Catalytic dehydrogenation of —. E. K. Rideal. Proc. Roy. Soc., 1921, A99, 153—162.*

IN confirmation of Sabatier's experiments the application of the Nernst approximation formula indicates that the variation of the dissociation constants of the equilibria,  $\text{CH}_3\text{CHO} + \text{H}_2 \rightleftharpoons \text{C}_2\text{H}_5\text{OH}$  and  $\text{CH}_3\text{COCH}_3 + \text{H}_2 \rightleftharpoons \text{CH}_3\text{CH(OH)CH}_3$ , with the temperature are extremely large. The velocity of decomposition at the surface of a solid catalyst is much higher than the reverse reaction of hydrogenation. The variation of the values of the dissociation constants with the temperature was experimentally determined with a constant volume thermometer containing reduced copper as catalytic material, and at low pressures a fairly close approximation to reversibility was obtained. The value of  $\Delta U$ , (heat of reaction) for the decomposition of ethyl alcohol was determined as 11,500 cal., whilst the mean value from the heats of combustion is 10,700. In the case of isopropyl alcohol the respective values are 21,300 and 11,800 cal., but an error of 1% in determining the heat of combustion of the alcohol would account for the discrepancy.

—G. F. M.

*Verbenene (dehydro- $\alpha$ -pinene) and certain of its derivatives. A. Blumann and O. Zeitschel. Ber., 1921, 54, 887—894.*

CRUNE *l*-verbenene (J., 1913, 498) readily yields a crystalline dibromide, m.p. 70°—72° C.,  $[\alpha]_D^{25} = +297.65^\circ$  in benzene solution, from which the parent substance is regenerated by reduction with zinc and acetic acid. Pure *l*-verbenene has sp. gr. 0.8866 at 15° C.,  $[\alpha]_D^{25} = -100.61^\circ$ , whilst the *d*-compound has sp. gr. 0.8867 at 15° C.,  $[\alpha]_D^{25} = +100.71^\circ$ ,  $n_D^{25} = 1.49800$ . The mode of formation, physical constants, and chemical behaviour of the compounds are in harmony with the view that verbenene is dehydro- $\alpha$ -pinene. Reduction with sodium and ethyl

alcohol converts *l*-verbenene into dihydroverbenene ( $\delta$ -pinene), b.p. 158°—159° C. at 762 mm., sp. gr. 0.865 at 15° C., 0.8625 at 20° C.,  $n_D^{25} = 1.4662$ ,  $[\alpha]_D^{25} = +36.52^\circ$ . (Cf. J.C.S., July.)—H. W.

*Potassium oxalates. Sabalitschka. See VII.*

PATENTS.

*Lobelia inflata; Process for obtaining the active ingredient of —. C. H. Boehringer Sohn. E.P. 145,622, 29.6.20. Cony., 2.9.14. Addn. to 145,621 (J., 1921, 195 A).*

THE active  $\alpha$ -lobeline hydrochloride present in the aqueous solution after the crystallisation of the  $\beta$ -lobeline hydrochloride, can be extracted by other chlorinated solvents such as carbon tetrachloride, trichloroethylene, or tetrachloroethane, instead of chloroform.—D. F. T.

*Butyl alcohol; Manufacture of secondary —. C. Weizmann and D. A. Legg. E.P. 161,591, 24.2.16.*

LIQUEFIED  $\beta$ -butylene is mixed with 75% sulphuric acid, with phosphoric acid, or with benzenesulphonic acid, either at ordinary temperatures under pressure, or at  $-10^\circ$  to  $-15^\circ$  C. under atmospheric pressure, and when the hydrocarbon has completely dissolved the liquid is distilled with steam, pure secondary butyl alcohol passing over.

—G. F. M.

*[Hydr]oxaldehydes and their derivatives; Manufacture of —. G. B. Ellis. From Soc. Chim. des Usines du Rhône. E.P. 161,679, 13.1.20.*

HYDROXYALDEHYDES are produced by the action of formaldehyde on a phenol or a phenol derivative in presence of a nitroso-compound, such as nitrosobenzene or nitrosodimethylaniline, and, if necessary, a catalyst. For example, vanillin is obtained by treating for 1—2 hrs. a solution, in 50 pts. of methyl alcohol, of 4 pts. of guaiacol, the nitrosodimethylaniline derived from 8 pts. of dimethylaniline, and 8 pts. of 40% formaldehyde, hydrogen chloride being continuously bubbled through. The product is diluted with water, the methyl alcohol distilled off, and the vanillin extracted from the aqueous residue with ether or benzene. The nitrosodimethylaniline is mostly transformed into *p*-aminodimethylaniline. (Reference is directed, in pursuance of Sect. 8, Sub-sect. 2, of the Patents and Designs Acts, 1907 and 1919, to E.P. 139,153; J., 1920, 527 A.)—G. F. M.

*Oxalic acid; Manufacture of — from carbohydrates. Valentiner u. Schwarz, G.m.b.H. G.P. 329,591, 20.12.13.*

SUGAR (100 pts.) is treated below 70° C. with a mixture of, e.g., 320 pts. of sulphuric acid, 300 pts. of nitric acid, and 380 pts. of water in the presence of salts of vanadium, molybdenum, or manganese, as catalysts, yielding 142 pts. of oxalic acid. The reaction is allowed to continue until the liquor is free from nitric acid, and the nitrous fumes evolved are absorbed by the mother liquor from a previous preparation.—L. A. C.

*Alkyliminodisulphonic acids; Production of —. Farbenfahr. vorm. F. Bayer und Co. G.P. 330,801, 4.8.18.*

BAAR salts of iminodisulphonic acid react with organic halogen compounds, such as methyl iodide, epichlorhydrin, or ethylene bromide, or inorganic acid esters, such as dimethyl sulphate, in accordance with the equation,  $\text{KN}(\text{SO}_3\text{K})_2 + \text{R.Cl} = \text{KCl} + \text{R.N}(\text{SO}_3\text{K})_2$ . The salts of alkyliminodisulphonic acids hydrolyse in the presence of acid in two stages, with the production first of alkyliminomonsulphonic acids and then of alkylamines. Potassium dihydroxypropyliminosulphonate,  $(\text{SO}_3\text{K})_2\text{N.CH}_2\text{CH(OH).CH}_2\text{OH}$ ,

is prepared by the prolonged action of epichlorhydrin on an aqueous solution of potassium iminodisulphonate at normal temperature in the presence of potassium hydroxide and subsequent evaporation of the solution; the product is stable in neutral or alkaline solution, but is decomposed by boiling with acids into sulphuric acid and dihydroxypropylamine. The preparation of potassium ethylenediaminetetrasulphonate, and of potassium methylaminomonomosulphonate and methylamine from potassium methyliminodisulphonate, are also described.—L. A. C.

**Alcohols and amino-alcohols of the quinoline series; Preparation of —.** Preparation of quinolyl ketones. Vereinigte Chini-fahr, Zimmer und Co., G.m.b.H. G.P. (A) 330,813, 8.3.18, and (B) 330,945, 11.6.18. (B) Addn. to 268,830 (J., 1914, 277).

(A) QUINOLYL ketones and quinolyl aminoketones are reduced to secondary alcohols by treatment in alcoholic solution with a metal of which the hydroxide possesses weakly acid properties, such as zinc or aluminium, and with an alkali ethoxide. Quinone,  $\text{C}_6\text{H}_4(\text{O})_2$ ,  $\text{N} \cdot \text{CO} \cdot \text{C}_6\text{H}_4 \cdot \text{N}$ , yields a mixture of quinine and quinidine; the former is isolated as the insoluble sulphate, and the latter is extracted with ether from the mother liquors after they have been made alkaline. Dihydrocinchonine,  $\text{C}_6\text{H}_7\text{N} \cdot \text{CO} \cdot \text{C}_6\text{H}_4 \cdot \text{N}$ , yields dihydrocinchonine and dihydrocinchonidine, both of which occur naturally together with quinine, and also the two other possible stereoisomers, of m.p.  $126^\circ \text{C}$ . and  $106^\circ \text{C}$ ., and  $[\alpha]_D^{25} = +88.5^\circ$  and  $[\alpha]_D^{25} = +48^\circ$  respectively, which up to the present have not been found in nature; the two latter are both converted to dihydrocinchotoxine by boiling with dilute acetic acid. Cinchotoxine,  $\text{C}_6\text{H}_7\text{N} \cdot \text{CO} \cdot (\text{CH}_2)_2 \cdot \text{C}_6\text{H}_4 \cdot \text{N}$ , 6-methoxyquinolyl-4-methylketone, and 6-methoxyquinolyl-4-piperidylmethylketone, yield on reduction respectively cinchotoxol, a yellow oil, 6-methoxyquinolyl-4-methylcarbinol, m.p.  $120^\circ - 121^\circ \text{C}$ ., and 6-methoxyquinolyl-4-piperidinomethylcarbinol, m.p.  $109^\circ \text{C}$ . (B) The use is claimed of esters having the general formula  $\text{R}' \cdot \text{CH}_2 \cdot \text{COO} \cdot \text{R}''$ , in which  $\text{R}'$  is an N-acylated basic residue instead of an alkyl group, as claimed in the chief patent. N-Benzoylhomocincholoipon ethyl ester condenses with quinoline-4-carboxylic acid ethyl ester at  $80^\circ \text{C}$ . in the presence of sodium ethoxide forming a  $\beta$ -ketone acid ester, which on boiling with hydrochloric acid yield a quinolyl ketone identical with dihydrocinchotoxine, the transformation product of dihydrocinchonine.—L. A. C.

**Morphine alkaloids and derivatives of barbituric acid; Preparation of compounds of —.** Ges. für Chem. Ind. in Basel. G.P. 330,814, 6.6.17. Addn. to 322,335 (E.P. 107,409; J., 1917, 979).

ANÆSTHETICS for use in inducing "twilight sleep" are prepared by the action of arylalkylbarbituric acids on morphine alkaloids, as described in the chief patent. Morphinephenylethylbarbituric acid, ethylmorphinephenylethylbarbituric acid, and codeinephenylethylbarbituric acid melt respectively at  $250^\circ$ ,  $87^\circ$ , and  $80^\circ \text{C}$ .—L. A. C.

**Iron-albumin compounds; Preparation of water-soluble —.** Bauer und Co. G.P. 330,815, 11.4.19.

An aqueous solution of alhumose is added to a solution containing ferric nucleate and a neutral salt of an organic acid, such as sodium nucleate, trisodium citrate, or potassium sodium tartrate, until a test portion gives with yellow ammonium sulphide no black precipitate of iron sulphide, even after several hours, but yields a clear bluish-green solution; the solution is then evaporated to dryness. Ferric nucleate is prepared as a flesh-coloured

precipitate by the addition of ferric chloride to sodium nucleate in aqueous solution.—L. A. C.

**Mercury sludge [obtained, e.g., in the manufacture of acetaldehyde from acetylene]; Recovery of mercury from —.** Consortium für Elektrochem. Ind. G.m.b.H. G.P. 332,202, 30.8.17.

THE water is removed by heating the sludge below  $155^\circ \text{C}$ .—L. A. C.

**Ferments, enzymes, alkaloids and other substances; Extraction of — from animal organs and plants.** Gehr. Schubert. G.P. 334,660, 23.12.17.

THE fresh or dried material is mixed with sufficient water to form later with calcium chloride a solution which remains liquid well below  $0^\circ \text{C}$ . The moistened material is frozen and thoroughly comminuted, and calcium chloride is then added, whereupon liquefaction occurs. The calcium chloride solution formed, containing the substance required, is separated from the residual mass and worked up. The calcium chloride may be precipitated from this solution by an excess of salt, and after centrifuging, the liquid is concentrated to the required extent. The process ensures a very thorough disintegration of the tissues and a complete extraction of the substance required, e.g., pepsin from sheep's stomach, active principles from various glands, etc.—J. H. I.

**Petroleum; Production of a preparation [embrocation] from crude —.** Chem. Werke Grenzach A.-G. G.P. 334,916, 4.2.19.

CRUDE petroleum, freed from its more volatile constituents, is treated with hypochlorous acid or salts thereof, and afterwards with feebly basic oxides, hydroxides, or basic salts. The hypochlorous acid combines with unsaturated constituents of the petroleum and prevents any irritating effect when the latter is employed as an embrocation in cases of horse scabies and similar affections. The added bases neutralise acid constituents and give the product the consistency of an emulsified salve readily liquefied on warming.—J. H. I.

**Alcohol and ether vapours.** U.S.P. 1,376,069. See I.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### PATENT.

**Photomechanical designs, pictures, or printing surfaces; Process of producing —.** F. Hausleiter. E.P. 139,472, 23.2.20. Conv., 19.8.14.

A NEGATIVE on a transparent support is developed with a developer which hardens the gelatin in the neighbourhood of the image. The unexposed parts are washed away, and the remaining image is stained with a suitable dye to increase its opacity to the light to which bitumen is sensitive, and is then coated with bitumen, exposed from the back and developed in the usual way.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

### PATENTS.

**Explosive.** B. F. Halvorsen. U.S.P. 1,375,588, 19.4.21. Appl., 4.12.19.

A NITRO-DERIVATIVE of urea is utilised as an explosive.—W. J. W.

**Explosive composition.** Explosive. F. Olsen. U.S.P. (A) 1,376,029 and (B) 1,376,030, 26.4.21. Appl., 22.5.20 and 15.1.21.

(A) AN explosive composition contains an inorganic

perchlorate, a nitro-hydrocarbon, a propellant powder, a non-explosive combustible substance, a chlorine-fixing material, and a material for regulating the flame-temperature. (b) A nitro-starch explosive is mixed with a propellant powder.

—W. J. W.

*Potassium perchlorate explosives.* Chem. Fabr. von Heyden A.-G. G.P. (A) 298,568, (B) 300,669, and (C) 303,330, 6.12.16.

(A) A mixture of potassium perchlorate, liquid hydrocarbons, and finely-divided iron, without a nitro-compound, gives an explosive which can be readily detonated. (a) By the addition of ammonium perchlorate to potassium perchlorate explosives, not containing a nitro-compound, the proportion of hydrocarbons may be increased. (c) The use of nitroglycerin in potassium perchlorate explosives may be dispensed with and cheapness thereby increased, by adding an appreciable amount of chlorate, exceeding the 0.2% present in commercial pure potassium perchlorate. The resulting explosive has a high degree of safety.—W. J. W.

*Guncotton; Process for stabilising* —. Riensberg. G.P. 299,039, 17.8.16.

THE water used for the purification of guncotton is heated in boilers which are separated from the stabilising vats, and is automatically supplied to, and drawn from, the vats.—W. J. W.

*Smokeless powders; Manufacture of* —. Claessen. G.P. 299,760, 15.10.14.

GELATINISATION of smokeless powders, containing nitrocellulose and less than 30% of nitroglycerin or a nitro-compound, is effected, without the aid of a volatile solvent, by incorporating them at a temperature below 100° C., and raising the temperature of 100° C. after organic materials such as resin, diphenylamine, paraffin, etc., have been added. To prevent these substances permeating the powder and retarding its rate of combustion, a protecting film may be produced on the surface of the powder by adding substances such as starch, tragacanth, or gum arabic, which are insoluble in the nitroglycerin or nitro-compound, before introducing the other organic materials.—W. J. W.

*Liquid air explosives; Manufacture of* —. Westfälische Berggewerkschaftskasse. G.P. 300,728, 7.1.16.

IN the preparation of liquid air explosives, cork meal constitutes a suitable ingredient, inasmuch as it can absorb 6.7–7 times its weight of liquid oxygen and does not consume much oxygen on detonation. Liquid air explosives containing cork meal retain their sensitiveness to detonation for a longer period than those in which the absorbent consists of wood meal.—W. J. W.

*Liquid air explosives; Manufacture of* —. B. Diamand. G.P. 304,300, 1.6.17.

LIQUID hydrocarbons or their compounds are absorbed by, or crystallised in, moss or peat, which is then impregnated with liquid air or oxygen. Both moss and peat retain their elasticity even after absorption of the hydrocarbons, and are therefore readily formed into cartridges.—W. J. W.

*Detonating compositions containing lead azide and nitro-compounds.* W. Eschbach. G.P. 305,100, 1.1.18.

INFUSORIAL earth, or powdered asbestos, is added to lead azide compositions. Explosives obtained by this means are especially suitable for detonating cartridges which have been steeped in liquid oxygen.—W. J. W.

*Gunpowder and smokeless powder; Manufacture of an explosive consisting of a mixture of* —. Claessen. G.P. 308,538, 5.1.18.

A MIXTURE of gunpowder and smokeless powder consisting of nitroglycerin and nitrocellulose is less hygroscopic and leaves a smaller residue on combustion than ordinary gunpowder; such an addition to gunpowder therefore ensures more regular combustion.—W. J. W.

*Chlorate explosives; Manufacture of granulated* —. Fürstlich Plessische Miedziankfabrik. G.P. 307,100, 29.11.17.

SULPHITE-CELLULOSE waste liquors are added to the other ingredients of chlorate explosives, the moist mass being subsequently forced through sieves and dried. The granular explosive thus produced has a high efficiency.—W. J. W.

*Signals; Mixtures for light and smoke* —. A. Lang. G.P. 307,640, 8.5.17. Addn. to 265,656 (J., 1913, 1169).

THE mixtures described in the original patent as suitable for exterminating plant pests may be adapted for use in signals by the addition of light- and smoke-producing substances, e.g., metallic powders, such as lead or zinc, or compounds of cadmium and arsenic. Such mixtures will burn even in water.—W. J. W.

*Explosive propellants.* O. Silberrad. E.P. 150,002, 11.8.20. Conv., 21.8.19.

SEE U.S.P. 1,349,983 of 1920; J., 1920, 676 A.

## XXIII.—ANALYSIS.

*Zinc; Volumetric estimation of* —. I. M. Koltzoff and J. C. van Dijk. Pharm. Weekblad, 1921, 58, 538–553.

THE mercury double thiocyanate method is found to be best. The reagent is prepared by dissolving 1 mol. of mercuric nitrate in 4 mols. of potassium thiocyanate, and is added in excess to the zinc solution. The whole is made up to a known volume and filtered, and the excess of reagent titrated in an aliquot part of the filtrate by means of mercuric nitrate, using iron alum as indicator. (Cf. J.C.S., June.)—S. I. L.

*Cobalt; Nitroso-R-salt, a new reagent for the detection of* —. H. S. van Klooster. J. Amer. Chem. Soc., 1921, 43, 746–749.

NITROSO-R-SALT gives an intense red coloration or precipitate with cobalt salts, by means of which 1 pt. of cobalt may be detected in presence of 200 pts. of nickel. The salt is prepared by the action of nitrous acid on sodium 2-naphthol-3,6-disulphonate. The test is carried out as follows: To 2 c.c. of a dilute solution of the salt to be tested (1.5–2.0 g. in 100 c.c. of water) is added 1 g. of sodium acetate and 2 c.c. of nitroso-R-salt solution (0.5 g. in 100 c.c. of water). The liquid is boiled over a small flame and after the gradual addition of 1 c.c. of nitric acid (sp. gr. 1.4) the boiling is continued for at least 1 min. A permanent change in colour towards red indicates the presence of cobalt. The colours produced by other metals are destroyed by the nitric acid. It is essential that the original solution should not be acid, for this retards and may prevent the formation of the red coloration. (Cf. J.C.S., June.)—J. F. S.

*Arsenic reduction tube; Electrically heated* —. K. Zwicknagl. Chem.-Zeit., 1921, 45, 418.

The tube of a Marsh apparatus is heated by means of a length of resistance wire wound round it and insulated with asbestos-paper. To ensure better contact of the gases with the heated wall of the tube the latter is flattened so that its cross section is about 2x14 mm.—W. P. S.

*Hydrogen ion concentration; Colorimetric determination of — in small quantities of fluid.* L. D. Felton. J. Biol. Chem., 1921, 46, 299—305.

Drops of indicator solutions are mixed on a porcelain plate with drops of the solution to be examined and with drops of known buffer mixtures respectively. (Cf. J.C.S., June.)—G. B.

*Protein analysis; Modification of the Van Slyke method of —.* P. Menaul. J. Biol. Chem., 1921, 46, 351—352. (Cf. J., 1911, 1135.)

The humin is precipitated with phosphotungstic acid instead of with calcium oxide. (Cf. J.C.S., June.)—G. B.

*Chlorine, sulphur, and phosphorus in organic substances; Determination of —.* A. Grégoire and E. Carpioux. Bull. Soc. Chim. Belg., 1919, 28, 331—335.

The method, which is designed for use with plant products, consists in digesting the material with concentrated nitric acid and a little potassium permanganate in a distillation flask, the exit tube from which passes through a short condenser. At its lower end the exit tube is connected with the absorption device, which consists of two inclined tubes, 18—22 mm. diam., and about 1 m. long, arranged crosswise and joined by a connecting tube. To improve the contact between the absorbent and the vapours, pieces of glass tubing are placed in the inclined tubes, each piece being drawn out at one end which projects into the end of the next higher piece of tubing. The sulphur and phosphorus remain in the distillation flask as sulphuric and phosphoric acids, whilst the chlorine distils off along with nitrous fumes, and is collected in water as hydrochloric acid. The three acids are then estimated in the usual way.—W. G.

See also pages (A) 423, *Benzene hydrocarbons in gas* (Krieger). 424, *Chlorides in petroleum* (Matthews). 426, *Absorbing power of charcoal* (Kolthoff). 432, *Calcium carbide* (Drathen). 435, *Copper in molybdenum ores* (Bonardi and Shapiro); *Tin in cassiterite* (Corti); *Bismuth* (Spurge). 438, *Critical temperatures of solution of fats* (Vanderelde); *Sulphonated oils* (Hart). 440, *Filtration of tanning solutions* (Parker and Terrell). 441, *Chrome tanning liquors* (Thomas and Foster); *Tannin analysis* (Baldracco and Camilla); *Sulphuric acid in leather* (Immerheiser). 442, *Soil organic matter* (Read). 443, *Sucrose* (Sázavsky; also Browne). 444, *Dry substance of sugar products* (Kalshoven); *Titration of boric acid* (Boeseken and Couvert); *Extract yield from raw grain* (Windisch and Kohlbach); *Malt analysis* (Keil). 445, *Sodium carbonate in milk* (Vanderelde); *Goat's milk in cow's milk* (Austen); *Tallow in lard* (Vitoux and Muttelet). 446, *Morphine* (Ganassini). 447, *Iodotannic syrups* (Ugarte); 2.4.6-Trinitroresorcinol (Einbeck and Jablonski).

#### PATENTS.

*Gas analysis; Apparatus and process for —.* E. K. Rideal and H. S. Taylor. U.S.P. 1,375,933, 28.4.21. Appl., 5.3.19.

See E.P. 134,243 of 1918; J., 1920, 23 A.

*Gas calorimeters [; Regulating flow of water to —].* J. H. Fairweather. E.P. 162,826, 11.2.20.

*Ultra-filters.* G.P. 334,063. See I.

#### Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

#### I.—GENERAL; PLANT; MACHINERY.

##### APPLICATIONS.

Brettell (Soc. Anon. les Ateliers Réunis). Pulverisers and crushing-mills. 14,499. May 25.

Broadbent, Claughton, Hamilton, Jack, and Smith. Clarification of waste lubricants etc. 14,607. May 26.

Burden. Reducing or disintegrating machines. 14,843. May 28.

Gregory. Extinguishing-fluids. 15,127. May 31. Minerals Separation, Ltd., and Williams. 14,812. See X.

Möller. Apparatus for separating suspended bodies from gaseous fluids etc. 15,118. May 31. (Ger., 31.7.14.)

Pohlmann. Method of operating refrigerating-plants. 15,200. June 1. (Ger., 4.6.20.)

Rhodes. Barrel filter. 14,486. May 25.

Rigby. Heating or cooling liquids or admixed solids and liquids in evaporative etc. treatment. 14,599. May 26.

Sturgeon. Centrifugal separators. 15,038. May 31.

Williams. Tunnel ovens. 15,120. May 31.

##### COMPLETE SPECIFICATIONS ACCEPTED.

2242 (1920). Conder and Vivian. Crushers with vibrating jaws. (163,750.) June 8.

2243 (1920). Conder and Vivian. Roller mills. (163,751.) June 8.

3895 (1920). Josse and Gensecke. Evaporation of liquors. (138,871.) June 1.

5558 (1920). Testrup, and Techno-Chemical Laboratories, Ltd. Evaporating or distilling apparatus. (163,793.) June 8.

5860 (1920). Fuller-Lehigh Co. Pulverising-mills. (139,511.) June 1.

7189 (1920). Soc. l'Air Liquide. Apparatus for carrying out exothermic chemical reactions under high temperatures and pressures. (140,089.) June 8.

8228 (1920). Ely. Rotary furnaces. (163,543.) June 1.

8247 (1920). Read. Pulverising-machines. (163,856.) June 8.

10,387 (1920). Vanneau. Grinding or crushing apparatus. (141,721.) June 1.

11,927 (1920). Grondal. Apparatus for lixiviating granular or pulverulent material. (142,493.) June 8.

18,646 (1920). Stumpf. Heating and purifying boiler feed-water. (146,522.) June 8.

23,317 (1920). Alexander (Best). See II.

31,616 (1920). Velten. Apparatus for separating materials of different specific gravity. (155,570.) June 1.

35,921 (1920). Soc. d'Utilisation des Combustibles Pulvérisés. Rotary drying-apparatus. (163,653.) June 1.

# PATENT LIST.

[June 30, 1921.]

6986 and 6988 (1921). Kennedy. Gyrotory crushing-apparatus. (160,463 and 160,465.) June 1.

## II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### APPLICATIONS.

Beasley, Stenning, and Minerals Separation, Ltd. Production of coal briquettes. 15,066. May 31.

Benjamin and Treneer. Purification of hydrocarbons. 15,080. May 31. (U.S., 4.6.20.)

Clabaut and Coureycour. Gas-generators. 15,248. June 1. (Fr., 11.6.20.)

Cunningham and Stern. Production of water-gas. 15,001. May 30.

Helps. Manufacture and distribution of gas. 14,605 and 14,725. May 26 and 27.

Illingworth. Coking coal. 14,676. May 26.

Klee. 15,458. See XXIII.

Maclaurin. Water-gas plant. 15,203. June 1.

Marshall. Retorts for treating carbonaceous etc. materials. 14,472. May 24.

O'Byrne. Recovery and preparation of peat, turf, etc. for fuel etc. 14,428. May 24.

Piron. Regenerative coke-ovens. 15,363. June 2.

Poore. Destructive distillation of wood etc. 14,820. May 27.

Rigby. Heat-regenerating systems of gas-producer plants etc. 14,598. May 26.

Shields (Mine Safety Appliance Co.). Gas-purifying compositions, and their production. 15,330. June 2.

Tully. Manufacture of gas for heating and lighting. 15,254. June 1.

Umpleby. Gas producers and/or retorts. 14,276. May 23.

### COMPLETE SPECIFICATIONS ACCEPTED.

6428 (1919). McLeod. Apparatus for carbonising and distilling moist carbonaceous materials. (163,343.) June 1.

18,151 (1919). Dalley (Greenway). Distillation or cracking of hydrocarbon oils. (163,347.) June 1.

28,614 (1919). Illing and Kelly. Treatment of petroleum etc. (163,363.) June 1.

6235 (1920). Williams, Francis, and Bynea Steel Works, Ltd. Gas-producers. (163,513.) June 1.

6509 (1920). Fyleman. Separation of mineral oils etc. from sand or rock. (163,519.) June 1.

8955 (1920). International Coal Products Corp. Manufacture of carbonised fuel briquettes. (140,821.) June 8.

17,613 (1920). Nasspress Ges. Utilising coal slimes. (146,264.) June 8.

19,151 (1920). Meyer. Carbonising solid fuels in a circulating current of gas. (147,117.) June 8.

19,308 (1920). Otto u. Co. Coke-ovens. (147,229.) June 1.

19,310 (1920). Otto u. Co. Coke-ovens. (147,231.) June 8.

19,512 (1920). Collin A.-G. Recovery of by-products from fuel gases. (147,633.) June 1.

20,608 (1920). Riedel. Recovery of nitrogen contained in fuel. (148,784.) June 8.

23,317 (1920). Alexander (Best, Inc.). Oxidation of finely subdivided material, and combustion apparatus therefor. (162,623.) June 1.

2833 (1921). Illing and Kelly. Production of asphalt from petroleum etc. (163,656.) June 1.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

### APPLICATIONS.

Bowler. Decorticating-machine and chemical apparatus for treating palm fibres. 14,374. May 24.

Brodie. Treatment of esparto grass, fibrous material, etc., for manufacture of paper. 15,205. June 1.

Budde, and Hendon Paper Works Co. Manufacture of hydro-oxy-cellulose, a new xanthogenated compound therefrom, etc. 14,852. May 28.

Dreaper. Spinning filaments of artificial silk. 14,256. May 23.

Dreyfus. Coating or treating surfaces etc. 14,802. May 27.

Dreyfus. 14,861. See IX.

### COMPLETE SPECIFICATIONS ACCEPTED.

999 (1920). Braun. Manufacture of cellulose. (137,831.) June 1.

3916 (1920). Fearnley. Manufacture of paper. (163,421.) June 1.

4693 (1920). Braun. Manufacture of cellulose. (139,171.) June 1.

4877 (1920). Bronnert. Manufacture of viscose silk. (163,466.) June 1.

5983 (1920). Bronnert. Manufacture of viscose silk. (163,817.) June 8.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### APPLICATIONS.

Calico Printers' Assoc., Ltd. Printing variegated effects on fabrics etc. 15,414. June 3.

Charlesworth. Dyeing processes. 15,482. June 4.

Silbereisen. Apparatus for continuous scouring, dyeing, etc. of fabrics in the piece. 14,441. May 24. (Fr., 2.3.21.)

### COMPLETE SPECIFICATIONS ACCEPTED.

5188 (1920). King and Haines. Bleaching and bleaching compounds. (146,078.) June 1.

14,820 (1920). Poulson. Sizing textile fabrics etc. (163,915.) June 8.

1343 (1921). Krantz. Dyeing yarn on bobbins in circulating dye liquor. (157,418.) June 8.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

### APPLICATIONS.

Casale and Leprestre. Apparatus for catalytic synthesis of ammonia. 14,459. May 24.

Elektrizitätswerk Lonza. Treatment of residuary ammonium chloride lyes. 14,900. May 28. (Switz., 29.5.20.)

Evershed and Gitsham. Manufacture of sulphate of lead. 15,078. May 31.

Griffith. Schlippe's salt etc. 14,920. May 30.

Heenan and Froude, Ltd., and Walker. Concentration of brine. 14,501. May 25.



Jourdan. Ammonia-soda process. 15,426. June 3. (Ital., 3.6.20.)

Wright and Wright. Production of ammonium salts and coloured pigments. 15,023. May 31.

#### COMPLETE SPECIFICATIONS ACCEPTED.

9994 (1920). Phillips. Manufacture of readily soluble alkali silicates. (163,877.) June 8.

15,458 (1920). Badische Anilin u. Soda Fabrik. See XVI.

20,568 (1920). Jaubert. Apparatus for purifying hydrogen or oxygen. (148,564.) June 1.

### VIII.—GLASS; CERAMICS.

#### APPLICATIONS.

Atkinson, and Stein and Atkinson. Drying china clay, etc. 15,304. June 2.

Deussing. Production of artificial meerscham. 14,592. May 25. (Ger., 7.6.20.)

Techno-Chemical Laboratories, Ltd., and Testrup. Manufacture of china clay. 14,992. May 30.

#### COMPLETE SPECIFICATIONS ACCEPTED.

5597 (1919). Gowen. Manufacture of refractory materials. (124,208.) June 1.

25,262 (1920). Festa. See X.

### IX.—BUILDING MATERIALS.

#### APPLICATIONS.

Dreyfus. Treatment of wood, paper, etc. 14,861. May 28.

Hawes. Substitute for wood etc. 14,782. May 27.

#### COMPLETE SPECIFICATIONS ACCEPTED.

26,280 (1919). Dale. Concrete etc. (163,731.) June 8.

4337 (1920). Mattison. Manufacture of fibrous cement products. (133,921.) June 1.

4398 (1920). Lewis. Continuous kilns for burning bricks etc. (163,439.) June 1.

4890 (1920). Brothers. Manufacture of plaster of Paris. (163,468.) June 1.

10,302 (1920). Milner and Robinson. Manufacture of bricks, blocks, etc. from blast-furnace slag. (163,569.) June 1.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

#### APPLICATIONS.

Alloy Welding Processes, Ltd., and Jones. Electrodes, welding rods, and soldering sticks. 15,435. June 3.

Aymard. Treatment of silica-bearing ores. 14,946. May 30.

Jones, and Sylvette, Ltd. Nickel alloys. 14,603. May 26.

Loke. Direct reduction of titanite-iron ores or slags. 14,813. May 27.

Marshall. Anticorrosive and antifouling composition for metals. 15,174. June 1.

Minerals Separation, Ltd., and Williams. Prevention of dusting of powdered minerals. 14,812. May 27.

Thompson (Aikens). Electrolytic refining of tin. 15,047. May 31.

Turner. Production of carbon-free ferromolybdenum. 15,179. June 1.

Wardall. Aluminium alloys. 14,544. May 25.

#### COMPLETE SPECIFICATIONS ACCEPTED.

19,015 (1919). Niiranen and Hibbert. Recovery or separation of metals. (163,348.) June 1.

7139 (1920). Irons. Manufacture of steel. (163,528.) June 1.

8468 (1920). Grondal. Apparatus for use in concentrating ores etc. (140,775.) June 1.

9136 (1920). Rustproofing Syndicate, Ltd., and Newman. Metal pickling or cleaning. (163,868.) June 8.

9250 (1920). Vermaes, and Syndicaat Electro-Staal. Manufacture of iron from iron ores. (163,561.) June 1.

10,302 (1920). Milner and Robinson. See IX.

17,393 (1920). Krupp A.-G. Production of low-carbon and low-silicon ferrochromium. (145,711.) June 8.

20,131 (1920). Zelewski. Zinc-extracting furnaces with vertical retorts. (148,210.) June 1.

20,181 and 20,200 (1920). Dagory. Removal of copper deposit from the bores of guns. (148,250 and 148,263.) June 8.

22,562 (1920). Lavandeyra. Aluminium alloys. (149,633.) June 8.

25,262 (1920). Festa. Furnaces for fusing steel and other metals, baking ceramic products, refractory materials, etc. (155,231.) June 1.

5614 (1921). Brown, Boveri & Co. See XI.

6417 (1921). Passalacqua. Soldering aluminium or aluminium alloys. (159,480.) June 1.

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Alloy Welding Processes, Ltd., and Jones. 15,435. See X.

Boite, Edridge, and Lyto, Ltd. Galvanic batteries or cells. 14,302. May 23.

Chloride Electrical Storage Co., Ltd., and Heap. Secondary electric batteries. 14,571. May 25.

Loke. Electric etc. furnaces. 14,814. May 27.

Philippart. Electric accumulators. 14,543. May 25. (Fr., 31.5.20.)

Rennerfelt. Electric furnaces. 14,702. May 26. (Sweden, 2.6.20.)

Thompson (Aikens). 15,047. See X.

#### COMPLETE SPECIFICATION ACCEPTED.

5614 (1921). A.-G. Brown, Boveri & Co. Electrically heated muffle furnaces. (159,195.) June 1.

### XII.—FATS; OILS; WAXES.

#### APPLICATIONS.

Bolton and Lush. Recovery of oil from and purification of fuller's earth, charcoal, etc. for re-use in oil refining. 14,413. May 24.

Krebitz. Separating saponaceous matter from lime sludge. 15,428. June 3.

COMPLETE SPECIFICATION ACCEPTED.

23,957 (1919). Bolton and Lush. Conversion of fatty acids or oils, or fats containing free fatty acids, into glycerides. (163,352.) June 1.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Clarke, and Cookson and Co. Manufacture of paints and enamels. 14,956. May 30.

Macpherson. Preparation of pigments from tinsiferous laterites. 15,303. June 2.

Pearson. Treatment of acaroid resins. 14,753. May 27.

Wright and Wright. 15,023. See VII.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Abrey. Plastic material made from casein. 14,943. May 30.

Carmichael and Ockleston. Tanning. 14,943. May 30.

Glover and Martin. Preparation of chrome tanning agent. 14,654. May 26.

COMPLETE SPECIFICATIONS ACCEPTED.

20,665 (1919). Serle. Production of casein cements. (163,349.) June 1.

5690 (1920). Melamid. Manufacture of artificial tanning substances. (147,534.) June 8.

13,006 (1920). Bayerische Maschinenfabr. Regensburg. Rotary tanning and fulling drums. (143,223.) June 1.

19,964 (1920). Korn. Production of a substitute for leather etc. (147,910.) June 8.

XVI.—SOILS; FERTILISERS.

COMPLETE SPECIFICATIONS ACCEPTED.

23,304 (1919). Wolf and Fry. Manures. (163,359.) June 1.

3637 (1920). Sams. Manner and method of making same. (163,417.) June 1.

15,458 (1920). Badische Anilin u. Soda Fabrik. Transforming ammonia into a salt for use as a fertiliser. (144,659.) June 8.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

Classen. Manufacture of sugar from wood etc. 15,249. June 1. (Ger., 4.6.20.)

Petree. Manufacture of sugar. 15,457. June 3.

COMPLETE SPECIFICATIONS ACCEPTED.

16,322 (1920). Patterson. Manufacture of syrups and sugar. (163,924.) June 8.

22,021 (1920). Tavroges, Roche, and Martin. Purification of crude lactose. (163,937.) June 8.

XVIII.—FERMENTATION INDUSTRIES.

COMPLETE SPECIFICATION ACCEPTED.

23,594 (1919). Meyer. Production of yeast. (144,244.) June 8.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Atkinson, and Stein and Atkinson. Destruction of refuse. 15,305. June 2.

Howles. Insecticides, fruit washes, etc. 15,413. June 3.

Nielsen. Sterilising milk. 15,136. May 31.

Torrance. Manufacture of chocolate or cocoa products. 15,514. June 4.

COMPLETE SPECIFICATIONS ACCEPTED.

5707 (1920). Haddan (Naaml. Vennoots. Algem. Norit Maatsch.). Treating large quantities of liquids with purifying reagents in a continuous manner. (163,505.) June 1.

8310 (1920). Horlick. Food compound. (140,472.) June 8.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Farbw. vorm. Meister, Lucius, u. Brüning. Manufacture of methylsulphites of secondary aromatic-aliphatic amines. 14,986. May 30. (Ger., 31.5.20.)

Goodyear Tire and Rubber Co. Process of making thioureas. 15,088. May 31. (U.S., 5.6.20.)

Peuffaillit. Industrial preparation of derivatives of bornyle for preparing synthetic camphor. 15,441. June 3. (Belg., 10.1.21.)

COMPLETE SPECIFICATIONS ACCEPTED.

9667 (1920). Imray (Soc. Chem. Industry in Basle). Manufacture of mercury compounds of glucosides. (163,874.) June 8.

15,621 (1920). Du Pont de Nemours and Co. Production of tetra-substituted ureas. (144,681.) June 8.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Davies. Manufacture of photographic papers. 14,549. May 25.

Griffith. 14,920. See VII.

XXII.—EXPLOSIVES; MATCHES.

COMPLETE SPECIFICATION ACCEPTED.

25,436 (1920). Lundsgaard. Explosives. (163,946.) June 8.

XXIII.—ANALYSIS.

APPLICATIONS.

Hedley. Gas combustion furnaces for organic analysis. 14,262. May 23.

Klee. Apparatus for determining flash points of oils etc. 15,458. June 3.

COMPLETE SPECIFICATION ACCEPTED.

14,621 (1920). Svenska Aktiebolaget Mono. Apparatus for use in analysing gas mixtures. (143,918.) June 8.

# JOURNAL OF THE SOCIETY OF CHEMICAL INDUSTRY

Vol. XL. No. 1

January 15, 1921

## REVIEW

### SOCIETY OF CHEMICAL INDUSTRY.

#### CO-OPERATION IN PUBLICATION.

The memorandum submitted by the Federal Council for Pure and Applied Chemistry for co-operation in the publication of chemical literature, which we have been allowed to bring before our members through the courtesy of our President, Sir William Pope, who is also the Chairman of the Federal Council, is one of great moment to this society.

The outstanding national importance of securing a full record of chemical literature so that we are no longer dependent upon the publications of other countries is recognised on all sides, and the need for co-operation is equally evident if this desirable objective is to be achieved. Such co-operation has been in the minds of many for a considerable period, but the initial difficulties in the way of its successful realisation, with due regard to the individuality of each of the Societies concerned, have hitherto proved a serious barrier. The conditions that have resulted from the war and the present economic pressure have accentuated the need from every point of view, and it is accordingly to be hoped that the present proposal will be considered from a wide and comprehensive standpoint and with a broad outlook towards the future of pure and applied chemistry in this country.

The memorandum, which was drawn up by the Federal Council at its meeting held on November 3, was accompanied by a covering letter suggesting that the Councils of the Chemical Society and of this Society should each appoint four representatives to meet as a Joint Committee to discuss the questions raised; this suggestion has been acted upon, and the representatives have been instructed to report the results of their deliberations to their respective Councils as well as to the Federal Council.

From the standpoint of this Society this proposal brings with it a welcome opportunity for a full consideration of the Society's publications. Our Journal is our great asset, and the Council and the Publications Committee fully realise their responsibility to make it as efficient as possible, and to maintain its high standard and reputation. The Transactions, the Abstracts and the Review represent a record of chemical literature which has proved of the utmost value and which must be maintained in any co-operative scheme of publication. For progressive development, responsible, informed and constructive proposals will always be welcomed, and we look especially to the younger members for help in this direction. Both in the interests of this Society and in the wider interests of the chemical industries of the nation, the improvement of our chemical literature and its more efficient organisation merit serious attention and wise counsel if we are to march successfully in the competitive advance of science and its technical applications.

#### ON CHEMICAL PERIODICAL LITERATURE.

(Memorandum from the Federal Council for Pure and Applied Chemistry.)

In past years the desirability of closer co-operation for publication between the several societies which issue chemical journals has often been discussed, but, although certain minor mutual arrangements have been made, no effective joint action has been taken. The main object has been to retain a high standard of publication and, in the absence of financial embarrassment, the extra cost of the luxury of independent publication has proved of quite secondary importance.

The recently established economic conditions have, however, introduced financial difficulties; our large chemical societies have had to raise their subscriptions and to abbreviate their publications. The two chief societies receive considerable financial assistance from their advertisements, which have notably increased during recent years; in fact, the Society of Chemical Industry draws a much larger income from advertisements than from the annual subscriptions of its members. With the disappearance of the Excess Profits Duty it is to be anticipated that advertisement matter will be more difficult to obtain; it is thus likely that further increases in the annual subscriptions will be necessary in the near future.

The whole subject of chemical periodical literature should therefore now be considered afresh in view of the incidence of economic pressure as a new factor. It seems necessary to consider whether our present method of dealing with the corporate interests of chemistry, as represented by periodicals, cannot be so modified as to ensure greater economy—that is, greater efficiency.

For the present it is perhaps convenient to restrict inquiry to the Chemical Society and the Society of Chemical Industry; at this preliminary stage the consideration of the circumstances of the many other societies having chemical interests will needlessly complicate the issues. The third large chemical society, the Institute of Chemistry, being a qualifying body, is so different in type from the other two that it also may be left out of immediate consideration. We may therefore consider in what way the incomes of the two large societies named above can be so expended as to yield a greater return to their members than is at present attained. As a preliminary it will be useful to note the practice in other countries.

In the United States, the American Chemical Society publishes original scientific papers in its monthly journal, which absorbed the *American Chemical Journal* some years ago, and also publishes monthly the *Journal of Industrial and Engineering Chemistry*, which contains ephemeral matter and market reports in addition to original papers on technical chemistry. This Society also issues *Chemical Abstracts*, a fortnightly publication which aims at abstracting every published article of chemical interest; it is now constructing a formula index to include inorganic as well as

organic substances with a view to incorporating this in its decennial index.

In Germany, efforts were being made before the war to gather control of all the chemical periodical publications into one set of hands. In 1897, the German Chemical Society, which previously published abstracts as a part of the *Berichte*, acquired the *Chemisches Zentralblatt* and has since used this for the publication of chemical abstracts; in 1919, the *Chemisches Zentralblatt* was divided into two parts, scientific and technical, which now appear separately.\* Later this Society secured to its members preferential rates of subscription to the *Zeitschrift für angewandte Chemie* and to *Liebig's Annalen* and, with its possession of the large compendia, such as "Beilstein" and "Richter," was evidently intending to concentrate all important periodic publications, academic and technical, under its own guidance.

In Holland, the *Nederlandsche Chemische Vereniging*, numbering about 1000 members, issues a monthly journal of original scientific papers, the *Recueil des Travaux Chimiques des Pays-Bas*, and a weekly journal of technical and general interest, the *Chemisch Weekblad*, which also gives market prices. The Society also publishes the *Chemisch Jaarboekje* in three parts. The first part gives particulars of the Association and a list of its members and officers, together with a classified and indexed list of Dutch firms concerned with chemical products or necessities, and states the specialities of these firms; the second consists of tables of constants similar to but smaller than Biedermann's *Kalender*. The third part forms a very extensive chemical bibliography stating in which of the Dutch libraries each journal or book is to be found.

In Italy, the *Associazione Italiana di Chimica Generale ed Applicata* publishes monthly the *Gazzetta Chimica Italiana*, a purely scientific journal, and also monthly the *Giornale di Chimica Industriale ed Applicata*, which contains technical, general, and commercial matter.

In France, the *Société Chimique de France* was until two years ago the only large chemical organization; recently, however, the *Société de Chimie Industrielle* has been formed and has become very influential. As yet these two societies are entirely unconnected; each publishes its own original papers and abstracts. The relations between the two French societies seem practically identical with those which subsist between the Chemical Society and the Society of Chemical Industry.

In this country the Chemical Society issues the following publications:—

1. Transactions of the Chemical Society.
2. Abstracts of the Chemical Society.
3. List of members of the Chemical Society.
4. Annual reports on the progress of chemistry.

The Society of Chemical Industry issues the following:—

5. The Transactions, consisting of original papers.
6. The Review, containing matter largely of transient interest, with reviews of books.
7. The Abstracts of original papers and patents.
8. List of members of the Society.
9. Annual reports on the progress of technical chemistry.

Numbers 5, 6 and 7 are published together in fortnightly parts but with separate pagination.

The last inquiry showed that of the 3000 Fellows of the Chemical Society, and the 4000 members of the Society of Chemical Industry only 1000 are common to both; it appeared that an overlap of

abstracts to the extent of about 15 or 20 per cent. occurred as between the two journals of abstracts, but it is probable that this is now reduced to little more than 5 per cent.

It is impossible to consider the above series of publications issued and the editorial establishments maintained by these two entirely independent societies without concluding that economies are possible. Further, the fact that information (ephemeral or popular interest only appears for nightly in the *Journal of the Society of Chemical Industry*, has led to the appearance of weekly journals under private control which convey such information more promptly and, in addition, gain support from the more frequent distribution of advertisement matter; this factor naturally depreciates the value of the journals of the societies as advertising media.

The consideration of the tendency of foreign practice and of the circumstances of publication by the Chemical Society and the Society of Chemical Industry leads to the conclusion that increased economy and efficiency would be attained if they amalgamated into a "Chemical Federation" for purposes of publication. As to what set of publications they should issue provides matter for detailed discussion, and it is now only tentatively suggested that they should aim at the production of the following series:—

1. Transactions of the Chemical Society.
2. Transactions of the Society of Chemical Industry.
3. Chemical Abstracts, scientific and technical, purporting to be a complete collection of abstracts of original papers and patents, together with a list of books, Government reports, and the like.
4. A joint annual report on the progress of pure and applied chemistry.
5. A list of members of the chemical profession with their addresses and an indication of the societies to which they belong.
6. A weekly journal, containing the proceedings and notices of both societies, general summaries, reports of meetings, notices of books, parliamentary and legal information, company reports, market prices, notes of appointments, letters from correspondents, and the like.

Issues 1 and 2 would probably be made monthly and in much their present form. Issue 3 might appear fortnightly and aim at being as complete as the American *Chemical Abstracts*; it would probably be divided into scientific and technical sections like the *Zentralblatt*, and should be completed annually by a formula index, somewhat on the American plan, which could be employed in the production of complete indexes to the literature. Publication 5 could be made an indispensable chemical directory, giving particulars about learned and technical societies, possibly also lists of chemical firms and the like; it should be possible to secure the co-operation in this production of all the societies connected with chemistry, such as the Institute of Chemistry, the Society of Public Analysts, etc., with a view to their using this as their annual list of members. It could be made more complete by including particulars of chemical companies similar to those given in the *Stock Exchange Year Book*; its wide circulation would make it a valuable advertising medium, and it might thus pay for itself.

The weekly publication 6 should be more valuable for advertising purposes than our present journals.

In carrying out such a scheme as the above it would be possible to allow each Fellow of the Chemical Society to subscribe for 1, 3, 4, 5, and 6, and each member of the Society of Chemical Industry for 2, 3, 4, 5, and 6; also to obtain the remaining publication of the Chemical Federation at a reduced rate in the manner practised in the German Chemical Society. It would be necessary

\* [Since the memorandum was drawn up, certain changes in the modes of issue of the *Zentralblatt* and the *Zeit. f. angew. Chem.* have been notified for 1921 (cf. J., Dec. 31, 1920).—Ed.]

to apportion the financial outgoings and incomings as between the two societies; this involves purely actuarial work which can be safely left to professional hands.

#### COMMITTEES OF COUNCIL AND REPRESENTATIVES ON OUTSIDE BODIES.

**Publications Committee.**—Messrs. C. A. Keane (convener), E. F. Armstrong, J. L. Baker, W. J. A. Butterfield, F. H. Carr, W. H. Coleman, S. H. Davies, W. R. Hodgkinson, G. T. Morgan, J. A. Reavell, W. F. Reid, and E. Thompson, with the following co-opted members: E. R. Bolton, C. F. Cross, J. T. Dunn, E. Grant Hooper, J. R. Partridge, W. J. Rees, and W. G. Wagner.

**Finance Committee.**—Messrs. E. V. Evans (convener), E. F. Armstrong, J. L. Baker, C. S. Garland, J. Gray, H. Louis, S. Miall, W. F. Reid, and E. Thompson.

**General Purposes Committee.**—Messrs. S. Miall (convener), F. H. Carr, W. R. Hodgkinson, H. Louis, G. W. Monier-Williams, G. T. Morgan, L. G. Radcliffe, A. Réé, and E. Walls.

**Technical, Research, and Allied Societies Committee.**—Messrs. J. Allan (convener), E. F. Armstrong, W. A. Bone, J. W. Cobb, A. Holt, D. S. Jerdan, C. A. Keane, H. Levinstein, H. Louis, F. R. O'Shaughnessy, J. A. Reavell, and J. H. Young.

**Government and Parliamentary Committee.**—Messrs. W. F. Reid (convener), John Allan, J. L. Baker, W. A. Bone, Sir John Brunner, Bart., C. S. Garland, Sir R. A. Hadfield, Bart., S. Miall, and A. Réé.

**Literary and Libraries Committee.**—Messrs. W. J. A. Butterfield (convener), A. Holt, A. Lauder, R. L. Mond, G. T. Morgan, H. D. Smith, H. Talbot, and S. R. Trotman.

**Emergency Committee.**—The conveners of the above six committees; the General Secretary to act as convener.

The following have been nominated by the Council to represent the Society on outside bodies:—

**Federal Council for Pure and Applied Chemistry.**—Dr. C. A. Keane, Dr. S. Miall, and Mr. F. H. Carr.

**National Physical Laboratory (General Board).**—Sir Wm. J. Pope and Prof. F. G. Donnan.

**Conjoint Board of Scientific Societies.**—Dr. A. Holt and Dr. C. A. Keane.

**Institute of Chemistry Standards Committee.**—Dr. A. Holt, Prof. A. R. Ling, and Mr. W. G. Wagner.

**British Empire Sugar Research Association.**—Prof. A. R. Ling and Mr. J. W. MacDonald.

**British Association Fuel Economy Committee.**—Mr. E. V. Evans.

**Industrial Alcohol Joint Committee.**—Dr. E. F. Armstrong and Mr. D. Lloyd Howard.

**British Engineering Standards Association.**—Aircraft Sub-committee on Chemicals: Prof. W. A. Bone; Sub-committee on Textiles: Mr. C. F. Cross; Sub-committee on Dopes: Dr. J. N. Goldsmith.

**Imperial Mineral Resources Bureau.**—Lead, Silver, Zinc, and Cadmium Sub-committee: Mr. H. N. Ridge; Aluminium, Magnesium, and Sodium Sub-committee: Dr. R. Seligman; Minor Metals Sub-committee: Mr. W. G. Wagner; Chemical Industries (including Potassium) Sub-committee: Mr. C. S. Garland; Publications and Libraries Sub-committee: Mr. W. J. A. Butterfield; Abstracts Sub-committee: Prof. H. Louis and Mr. T. F. Burton.

#### NEWS FROM THE SECTIONS.

##### SHAWINIGAN FALLS.

The inaugural meeting was held on December 13, and was preceded by a dinner at which about sixty members and their guests were present.

The chairman, Dr. F. W. Skirrow, outlined the steps that had been taken to initiate the new Section, and read a cablegram from the President and Council wishing the Section every success on the occasion of its first meeting. Mr. G. MacIntyre then spoke and conveyed the good wishes of the Montreal Section.

The Hon. Walter Mitchell, Provincial Treasurer, addressed the meeting on the Province of Quebec, outlining its remarkable progress in regard to industry, transportation, and agriculture during recent years. The area of the Province, however, was so great—one-quarter that of the United States and five times that of the United Kingdom—and its natural resources so vast that its development had hardly begun. The Gouin administration had greatly increased expenditure on good roads, education, agriculture, and public works, but in such a judicious manner that the *per capita* debt had actually decreased during the past few years, a phenomenon unique in recent political history.

Development of the water-power resources by private capital had been encouraged and assisted by the Government, which even derived a profit from it, whilst the companies were able to sell power at very low rates. Of the 19 million available horse-power in Canada, Quebec possessed 685 million h.p., and of this \$75,000 h.p. had been developed. The Gouin dam at La Loutre, which cost \$2,500,000, and was serving an area of 300 sq. miles and impounding 160 billion cu. ft. of water, or twice as much as the Assouan dam in Egypt, had doubled the available horse-power on the St. Maurice River. In consequence, the large industrial towns of Grand'Merc and Shawinigan Falls had been built, and the village of Three Rivers had become a thriving city. Possible developments in other parts of the province were being studied by the Government. In concluding, the speaker pleaded for economy, co-operation between capital and labour, good sense, and good engineering in the difficult times now upon us, and predicted a period of prosperity greater than had been experienced in the past.

Mr. T. H. Wardleworth welcomed the new Section into the brotherhood of the Society which, he said, was not merely British, but imperial in character; as a vice-president, he was pleased to be able to endorse the cabled greeting from the President and Council in London. Great changes had taken place in the application of science to industry during the speaker's life-time. The period of "trial and error" had given way to insistence upon exact methods based upon well-ascertained facts, so that the chemist could reasonably anticipate results not only from laboratory experiments, but from the industrial development of new processes. Referring to the great care which Faraday bestowed upon his experimental researches, it was observed that although his mechanical and physical equipment was crude, his theories were nevertheless corroborated when modern refinements became available. Electricity had supplied the chemist of to-day with new powers and, in consequence, marked advances would be expected from a centre such as Shawinigan; it behoved every member of the new Section to see that these expectations were realised. Dealing with the relation of capital to chemical industry, the speaker emphasised the importance of the capitalist, and remarked upon the lack of education of wage-earners as revealed by thriftless habits. An appeal was made for a better understanding between capital and labour, as the vital interests

of both depended upon active co-operation. The labour problem was not always a question of hours and wages, and this was especially true in a country like Canada which employed cosmopolitan labour; due allowance must be made for national customs and habits.

After a vote of thanks to the speakers had been moved by Mr. Stadler, seconded by Mr. Wither-  
spoon, and replied to by Mr. Wardleworth, the meeting adjourned.

#### LONDON.

At the meeting held on January 3 at Burlington House, Mr. Julian L. Baker presiding, a paper by Mr. G. H. Thurston on "The Smith Continuous System of Carbonisation" was read, in the author's absence, by Colonel Brentnall. The carbonising process is carried out in two stages. In the first, coal of almost any description, after being dried with hot waste gases and crushed, is heated at 480° C. for 2 hours in cylindrical retorts lined with carborundum, the tar oils and gases being collected. The capacity of each retort is one ton of coal per hour, and mechanical stirrers are used to mix the charge and to move it forward from the inlet to the point of discharge. A coal with 35 per cent. volatile matter gives 5600—6700 cb. ft. of gas with calorific value 650—700 B.Th.U. The solid residue ("semicarbocoal") is briquetted with pitch and then heated for 6 hours at 1090° C. in inclined retorts; heavy oil, pitch, and ammonia are recovered from the volatile products, together with gas of 350—400 B.Th.U. "Carbocoal"—the residual solid—is stated to be an excellent smokeless domestic fuel, and also to be well suited for metallurgical, *e.g.*, blast-furnace, purposes, its crushing strain being 940 lb. per sq. in. The yields obtained from one ton of coal are:—14 cwt. of "carbo-coal," 10,030 cb. ft. of gas, 28 impl. galls. of tar, and 22.24 lb. of ammonium sulphate. The U.S. Government has assisted in developing the process, and two plants are now in operation, one at Irvington, N.J., producing 100 tons of "carbo-coal" per day, and the other, at Clinchfield, Virginia, having a daily output capacity of 500 tons; the former has been working for two years and the latter since July, 1920. Over one hundred different kinds of coal have been successfully treated at Irvington, and although coals containing 30 to 35 per cent. of volatile matter are the best, quite inferior grades have been found to give satisfactory results.

Dr. Irvine Masson and Mr. T. L. McEwan contributed three papers on "The Recovery of Solvent Vapours from Air." In examining the processes of Bréguet and others, the authors have carried out a large number of determinations of the solubility of ether and alcohol vapours in cresol and other solvents. From the data obtained they have deduced a mathematical expression which correlates absorption with temperature and the vapour pressures of the solvents; and have shown how the results can be used to calculate the amount of liquid solvent and the number and dimensions of the absorption towers required in large-scale practice. The solvent vapours are best recovered from solution in cresol by steam distillation. Ether vapour may be absorbed with sulphuric acid of over 75 per cent. concentration, and recovered by distillation. Alcohol vapour is readily absorbed by this strength of sulphuric acid, and liberated by distillation after adding water to dilute the acid to 50 or 40 per cent. concentration. Cresol was found to be more efficient than sulphuric acid as a solvent for alcohol and ether, and by its use large quantities of these liquids were recovered during the war. The third paper dealt with the analysis of ether, alcohol and water mixtures.

#### PRESENT AND FUTURE POSITION OF THE CHEMICAL SOCIETIES OF GREAT BRITAIN.\*

H. DROOP RICHMOND.

In 1914 the chemical societies were in a state of gentle activity, progressing slowly without any marked effort and reflecting the position of British chemical industry. The outbreak of war came as a sudden shock and revealed the facts that we had been dependent on other countries, notably Germany, for a very large portion of our chemical products, and had surrendered the position that we might have occupied. The reasons for this belong to the past, and any discussion of them opens such a wide question and has so little bearing on my subject that I simply record the fact; it is also a matter of recent history that British chemistry rose to the occasion, made up some of the leeway, and is now in the position it should have been many years ago. We have under the stress of necessity and at a great cost worked out for ourselves the fundamental principles of chemical manufacture of a large number of products which we did not make ourselves and have gained a knowledge which is approximating to that possessed by our competitors at the time when we commenced. We are not yet in a position to stand alone, for if we have learnt, so have they, and it will require more strenuous effort before we can compete in the world's markets.

The chemical societies helped in the national awakening, though under the conditions of stress the efforts made were largely individual and by groups, rather than by the chemists as an organised body; these conditions have ceased, and under the new circumstances we have time to work together to a common end, and the chemical societies can now take a large share in the advancement of British chemistry. Before the war there were four main bodies devoted entirely to chemistry:—The Chemical Society, for the advancement of pure chemistry; the Society of Chemical Industry, for that of applied chemistry; the Society of Public Analysts, for analytical chemistry; and the Institute of Chemistry, for the advancement of the profession of chemistry; and numerous other bodies, such as the Royal Society, the Royal Society of Arts, the British Association, and the Royal Institution, which promote the advancement of science including chemistry, and others, such as the Iron and Steel Institute, the Institute of Metals, the Biochemical Club, the Pharmaceutical Society, the British Pharmaceutical Conference, the Institute of Brewing, and many others which promote interests including more or less chemistry. Each society worked independently, though, partly from the fact that they had interests in common and partly because the prominent members of one body were often equally to the fore in the others, there was a certain amount of co-operation. The effect of the national crisis on the societies was, first of all, stagnation; then, as the outlook became clearer, the organisations of the societies were adapted to the national service, and each society has introduced changes to grapple with the altered economic conditions. It will be interesting to discuss those of the four purely chemical societies as indicating their present position.

The Chemical Society, which though localised in London is really a British society, has enlarged its scope to include the women chemists who helped to carry on during the war, and has initiated a scheme to render its library more generally available to all chemists.

The Society of Chemical Industry, which was the only body having local sections, has developed these and several new sections have sprung into being.

\* Paper read before the Nottingham Section on November 24, 1920.

notably in the Dominions and has some claim to be an Empire society. At the same time it has developed its central organisation in London, where many matters are dealt with without reference to its Sections, and has started an innovation in society journalism by publishing an ephemeral review of current chemical topics. A chemical engineering group has been formed.

The Society of Public Analysts innovated a very useful section of "Notes" in its journal, but has not otherwise made any alteration. This body, though small and of restricted scope, is a British society and has always included a large proportion of provincial members on its council.

The Institute of Chemistry has undergone a remarkable change. Always a qualifying body, it has enlarged the scope of its qualifications very greatly, a very large number of members has been admitted without the examination formerly insisted on, and, curiously enough, at a time when a house primarily designed as an examination hall has been built has permanently relinquished a not inconsiderable proportion of its examination tests. Local sections have been started in many parts of the country, and these local sections have unofficially enlarged the scope of the activities of the society by discussing problems in chemistry as distinct from professional chemistry. It has started a Journal, an enlargement of the former Proceedings.

The enlargement of the activities of the Institute has been brought about by the formation of the British Association of Chemists, which threatened at first to engulf it, but which has now become a body primarily promoting the material interests of those chemists who are engaged in process and similar work. There is also the National Union of Chemical Workers, a trade union doing very useful work, and the British Association of Chemical Manufacturers, whose chief function is chemical policy, and the Federal Council has been formed to unite the whole of the societies for one common object.

The financial pinch has been felt by all the societies, and two years ago the Institute of Chemistry took the bold step of doubling its subscription to Fellows and students and increasing it by 50 per cent. to Associates; this step was not wholly brought about by war conditions, but was caused partly by the expiration of the lease just before the war and the greatly increased cost of the new premises; the increase was probably greater than was absolutely necessary, and gives a surplus devoted to increased activities.

The Society of Chemical Industry and the Chemical Society are now following suit with increases of 66 per cent. and 50 per cent. respectively, accompanied in the case of the latter by a 25 per cent. reduction in the entrance fee.

The Society of Public Analysts has not yet made any increase.

All the societies have made economies, the most noticeable being the suppression of the list of members usually issued annually, and the discontinuation of authors' copies by the Society of Chemical Industry, and generally each society has economised in minor matters.

The Chemical Society and the Society of Public Analysts have made few obvious failures, but in my opinion both the Institute of Chemistry and the Society of Chemical Industry are open to criticism. I have already stated my views before the Institute of Chemistry (*cf. Proc. Inst. Chem.*, 1920, Vol. 2, p. 87), and in my opinion the Society of Chemical Industry spends too much on its central premises, and the Review portion of the Journal has not justified itself; as an ephemeral journal it appears with news too late and too trivial to meet the situation; the articles are often too weak, and it consists far too much of extracts from other papers on subjects not of great importance and in-

terest to technical chemists. The greatest failure of all is common to the three societies that publish journals—the continual overlapping of matter published in chemistry.

British chemical science needs the societies, needs a journal of pure chemistry, a journal of technical chemistry and one of analytical chemistry, either combined with the technical journal or independent, a standard up-to-date lexicon of chemistry, a chemical directory, a complete and easily available chemical library, and an ephemeral journal. It has been suggested that the National Exchequer should be asked to provide funds for some or all of these objects, but apart from the public clamour against increased expenditure, it must be remembered that Government aid means Government control and red-tape, and I doubt whether the objects aimed at would not be hampered by this. The advancement of chemical knowledge has been the work of chemists in the past, and must remain so, and it is only chemists themselves who can do the work. We have managed to find the time and the money so far and must do so in the future; chemistry has been built by each man doing his bit and the more bits that each can do the less money is required, and there are many men who would do a bit or a larger bit if they could be fitted into the machine; as it is, many of us duplicate the work that others are doing.

I am a whole-hearted advocate of amalgamation; I see the greatest difficulties in bringing this about, but I do not see any that are unsurmountable if we all make up our minds. I think that the financial stringency is leading us to see more clearly that a fusion of interests makes for economy and avoids inefficient expenditure of our chemical resources; the avoidance of the duplication that now goes on will mean considerable saving in publishing expenses, our heaviest financial encumbrance.

All the societies are centralised in London, and it is doubtful if London has the same pre-eminence as the centre of chemical thought and industry as it has as the national capital, and I feel convinced that to centralise our chemical societies there results in a loss of chemical power which might be made available if the large provincial centres were to take a more active part. The Society of Chemical Industry and now the Institute have recognised this by local sections, which are in a very healthy state, and the Chemical Society and the Society of Public Analysts need the same organisation; chemists want opportunities to meet and thereby gather a wider outlook and see the problems in a different light. There is a tendency as a society grows to leave the routine work which was done in the early days by voluntary effort to salaried officials, and the more efficient the official the more he wants to do and the more important he tries to make his post; and as his view necessarily cannot be that of the members at large he will frequently branch off into spheres of activity which do not yield the best results to the members and which add to the expenditure. He is frequently supported by the older and more prosperous members of his council, who see the advantages partly from the point of view of their very capable official, and who are not obsessed with the constant idea of economy. The older men also have lost some of their pristine energy, and it will be to the advantage of the societies generally to have a proportion of young energetic men who will realise more truly than the leaders of the profession the needs of the members to leaven the ripe experience of the elder men.

Chemists have now fully come to the limit of what they can pay for the objects for which the societies were founded, and unless expenditure is watched carefully their work will not progress; they have not, however, yet come to the limit of the voluntary service they will give, especially if it is recompensed



not only by experience gained but in addition by "kudos." There has never been any lack of persons who will take the honorary appointments, and I doubt if any chemist seeks payment for the original articles published under his name, and I believe many members would undertake voluntarily the work of local correspondents, indexers, contributors to a lexicon or bibliographers if the opportunity were given them. Purely clerical work, which must be done on the spot, editorial and journalistic work at which the amateur cannot hope to vie with the professional, and advertising (which is a profession by itself) must obviously be paid for, but much more voluntary work can be done than at present.

Since I wrote this Sir William Pope has sent me a letter which the Federal Council has addressed to both the Chemical Society and Society of Chemical Industry, and has given me permission to make use of it. Of the six publications suggested, the first three are obvious, and the fifth is one that I have already advocated in an even more extended form: I would only advocate that an editor with full powers should be appointed, who should have complete control of the journals, and that the publication committee should act only as consultant on questions of policy, leaving questions of detail to the editorial staff. About the annual reports I am not quite so convinced. Those of the Society of Chemical Industry are not in sufficient demand to make them pay, and I think the question as to their necessity should be considered very carefully. Personally, I read them with a very great interest, but I make no practical use of them. As to the weekly journal, this must be run on journalistic lines by a chemist and journalist combined and would compete with existing journals. My opinion is that it would be better to enter into an arrangement with one or more of the weekly trade journals rather than fight them.

The letter referred to modifies the propositions that I intended to make, and therefore I propose: (1) That the Society of Chemical Industry should forthwith accept the invitation of the Federal Council and nominate a representative of each of the Sections to consider the publication question; (2) that the representatives be empowered to offer the sectional organisations to other societies with a view to giving provincial members a more active part in the advancement of chemical knowledge; (3) that until joint publication is adopted the Society should purge its journal of the weak matter now published, include far more of the sectional proceedings, and strengthen the Publication Committee by adding elected representatives of every Section.

## THE BRITISH SCIENTIFIC GLASS INDUSTRY.

EDWARD QUINE.

The Committee on Commercial and Industrial Policy, under the chairmanship of Lord Balfour of Burleigh, in its Interim Report on Certain Essential Industries stated that:—"There are certain special commodities which are essential to national safety as being absolutely indispensable to important British industries, and were supplied before the war entirely or mainly from enemy sources or from sources under present enemy control"; and in submitting recommendations in connexion with these industries the Committee further stated that:—"A group of industries in the case of which very special treatment is necessary is that of the optical glass and instruments and the

chemical and laboratory glassware trades. The types of manufacture dealt with lie in the higher grades of the glass industry. To be successful they require specially skilled operatives and a direction not only commercial but possessing considerable scientific attainments. They carry meagre possibilities in direct profits, and as a result have not hitherto interested manufacturers in a degree in any sense commensurate with their importance in the industrial progress of the nation. Although comparatively small as regards the amount of capital required to be invested and of the labour employed therein, these industries are of vital national importance both on naval and military grounds and for all purposes of applied science and scientific research."

**Optical Glass and Scientific Instruments.**—Prior to the work of Schott and Abbe, a large portion of the optical glass required by British scientific instrument makers was made in the country, but the impetus and lead given by the researches of those scientists, which were supported by the Prussian Government, placed the German optical glass industry in such a commanding position that in 1914 only Messrs. Chance Bros., of Smethwick, were manufacturing optical glass in this country, and 80 per cent. of the optical glass used by British instrument makers, whose production was exceedingly small, was supplied by Germany.

The dependence of the safety of the nation upon the production of optical glass is clearly evident when it is appreciated that upon such glass depends the supply of range-finders, directors for artillery dial sights, gun sights, telescopes and binoculars, aeroplane-sighting and photographic instruments, trench and submarine periscopes.

Our position at the commencement of the war was truly alarming, and our armies took the field seriously handicapped owing to the predominance of our enemies in all instruments which may be termed the "eyes of the army."

During the war, although handicapped by shortage of skilled labour, by lack of materials, and by lack of knowledge of the manufacture of certain types of optical glass, British manufacturers and scientists laboured so successfully that before its termination practically every requirement of the scientific instrument maker, whether his products were required for the Forces or the laboratory, could be met by British optical glass.

To-day England can supply optical glass to meet all requirements of the British Empire in addition to those of foreign countries, and is producing certain types of optical glasses superior to those made in any other part of the world. The credit of this achievement is mainly due to the efforts and sacrifices of Messrs. Chance Bros., Messrs. Derby Crown Glass Co., Ltd., the late Mr. A. S. Fawcett, Sir Herbert Jackson, and Dr. C. J. Peddle.

**Chemical and Laboratory Glassware.**—Chemical and laboratory glassware is that glassware which is used in laboratories for scientific purposes, and may be divided into two main classes:—(1) Hollow glass ware, which is mainly manufactured at the glass furnace, e.g., flasks, beakers, petri dishes, etc. (2) articles of glassware which are made mainly from tubing at the blow-lamp flame, e.g., thermometers, pipettes, X-ray tubes, etc.

It will be readily appreciated that the dyestuff steel, chemical, explosive, gas, and metal industries require an adequate production of scientific glassware, as do also medical and research laboratories attached to hospitals, county and municipal laboratories devoted to public health and hygiene, and various laboratories at universities, colleges, etc. In fact, it may be fairly claimed that the scientific glass industry both in times of peace and war is the master "key" industry.

This industry was almost entirely in the hands

Germany and Austria before the war, and practically the whole of the hollow-blown furnace-made glassware was obtained from these countries. It is almost equally true that the entire lamp-blown scientific glassware was obtained from foreign countries, although there existed in this country a small number of highly skilled workers at the blow-lamp flame who produced the unrivalled English clinical thermometer and a limited number of types of chemical thermometers. In addition to these workers, there was a number of lamp workers who repaired damaged foreign glass apparatus, produced special lamp-blown glass apparatus, and tided over the difficulties of importers awaiting shipment of foreign-made glassware. These highly skilled glass workers could almost be counted on the fingers and belonged to the industrial class covered by the term "out-workers," whose premises were unpretentious and whose capital and apparatus were limited to a few pounds and a blow-lamp. It was fortunate that the dealers in laboratory glassware had large stocks when war broke out; otherwise the production of munitions would probably have come to a very sharp end, and it was soon apparent to those familiar with this industry that should the war be a long one the situation would become exceedingly critical. Steps were taken to produce scientific glassware in this country, but this was an exceedingly difficult task, for though it was possible for the chemist to analyse glassware and evolve formulæ, the commercial manufacture of the glass presented enormous technical difficulties. With no previous experience of this industry to guide them except that of the domestic glassware and the bottle industry, our glass manufacturers tackled the problems. In their endeavours they met with reverse after reverse, for, apart from a lack of knowledge, inadequate materials and glasshouse equipment, there was no British labour trained to blow the light hollow ware required in laboratories. Stocks rapidly diminished, manufacturers were pressed for their production, and British-made scientific glassware was placed on the market in a condition which was as unsatisfactory to the manufacturers as to the users, and could only be termed "stop-gap." Steadily, however, and at the cost of great monetary loss, the production of scientific glassware was improved, and to-day our manufacturers are producing light hollow-blown scientific glassware which is superior in resistance to chemical reagents, and general utility, to that produced in any other country.

The lamp-blown section of the industry is dependent upon an adequate supply of glass tubing, and before the war there were few skilled tube-drawers in this country, and although efforts were made to train workers, this was an undertaking requiring time, for the operation is one requiring great physical endurance and manipulative skill; and in spite of exceedingly high wages, even at the close of the war the number of skilled tube-drawers in this country was lamentably small.

The skill required of the operator who manufactures a lamp-blown article from tubing varies from that required to make a simple article, such as an ampoule, to that required for making a high-grade thermometer or X-ray tube, and it was therefore possible to increase rapidly the number of workers engaged in the simpler operations; but the high degree of skill required, together with the demands of the recruiting authorities, prevented any large increase in the numbers of the highly skilled workers. Yet on this weak section of the industry, struggling against innumerable difficulties due to lack of adequate glass tubing, lack of manufacturing plant and lack of skilled labour, was thrown at one critical period of the war a responsibility in connexion with our anti-submarine campaign on the satisfactory discharge of which our national

safety depended; and the glass-tube drawers and lamp-workers did not fail.

The demand for scientific glassware in the future will exceed that in the past, for it is obvious that the industrial progress and health of a community is becoming increasingly dependent upon its laboratories. Our Empire cannot afford to allow its industries, its health, and its safety in time of war to be dependent upon a commodity, the manufacture of which is controlled by a foreign nation.

We are, however, swiftly drifting into that position, for, owing to the difficulties experienced by manufacturers in developing the scientific glass industry, the high costs of building and equipment, and the strength and reputation of the German and Austrian manufacturers, the industry was never in a position to stand unaided against continental competition, and the rate of exchange has now made the position, without protection, hopeless.

The Standing Committee on Trusts, in its interim report just issued, says:—"The manufacturers of scientific glassware have incurred considerable losses since they undertook to manufacture this class of glassware, and although we are not able to report as to the fairness or unfairness of past and present prices, we consider that such temporary support should be afforded to this new industry as is consistent with fairness to consumers. If this were done, probably within reasonable time the industry would be able both to hold its own against foreign competition and to sell its productions at prices acceptable to the public."

Unrestricted importation of scientific glassware, however, is to-day crushing the British industry, and some manufacturers have been forced to cease production, others have discharged labour trained at great expense in the time of the nation's need, and soon, unless help in the way of preventing unfair continental competition is given, the industry will pass to Germany and Austria, and once more the Empire will depend upon our late enemies for that production which is most essential to our industrial progress and national safety.

## REPORT OF THE RAILWAY RATES ADVISORY COMMITTEE.

A. J. MALACRIDA.

(Transport Manager, Association of British Chemical Manufacturers.)

The Railway Rates Advisory Committee after sitting in public for forty days has now presented to the Minister of Transport its report (Cmd. 1098, 9d.) on the principles which should govern the fixing of tolls, rates and charges for the carriage of merchandise by freight and passenger train and by other services. As the report is very comprehensive, consisting of 54 foolscap pages, it is not possible to consider all the important points it raises, but as many of the proposals are of vital importance to the chemical industry, it is essential that these should be treated at some length.

The two main principles advocated by the Committee in respect of the fixing of future rates are:—

(1) The charge must in no case be less than the cost to the railway company of rendering the required services.

(2) The charge must in no case be more than the value of the services to the trader.

This seems a very sound policy, especially so far as the chemical industry is concerned, because at the present time the rates which are charged for the conveyance of chemical commodities are out of all proportion to the cost to the railway companies and

to the value of the service to the trader. It is not necessary to labour this point because it has already been fully dealt with in the articles which have appeared in previous issues of this *Journal*, by Mr. J. Lukes, traffic manager to Messrs. Brotherton and Co., Ltd.

In regard to the component parts of the new rates, the report states that all parties seem to be agreed that the present system should be continued, i.e., there should be a rate for conveyance and separate rates for station accommodation and services rendered at the terminals at each end of the transit, and that owners of private sidings should only be charged for the services actually rendered to them, the proper rates being fixed for terminal charges at the same time as the conveyance rates. The Rates Advisory Committee recommends that a new tribunal should be set up to deal with the fixing of the rates, tolls and charges, and to the present writer this appears to be a great step forward, which should prove of much benefit to the trading community as a whole. At the present time the fixing of rates and charges is left entirely in the hands of the railway companies, so long as they do not exceed their maximum power and do not create undue preference in favour of one trader as against another. Apart from this the trader has no voice in the fixing of the rates and charges, and the recommendation now made should assist in the development of industry generally. To illustrate this one might instance many cases in which representations have been made to the railway companies that certain commodities could not bear the rates charged between particular points, and in which the companies have refused to grant reductions on the ground that although the traffic would not pass between the particular points in which a trader was interested, it would pass in other directions. In future, cases of this sort would be brought before the new tribunal, which would doubtless consider them from all points of view, and in all probability it would grant such rates as would enable traffic to move freely; and whilst perhaps the rate might be on a lower basis than the railways would usually approve, it might still be remunerative, having regard to the possibility of developing the industry and increasing the traffic put on the railways.

From the standpoint of the chemical industry, the most important feature of the suggested tribunal is that it would decide as to which articles are to be classed as "dangerous goods." The wording of the recommendation is perhaps not quite as clear as the industry would desire, yet it is thought that the suggestion is a great advance on the existing position. It is well known that at the present time the railway companies have absolute power under Section 105 of the Railway Clauses Consolidation Act, 1845, to specify which goods are dangerous, and this decision, if based on the *bona fide* judgment of the railways that the goods are dangerous, cannot be altered except by a repeal of the Statute. This is aptly illustrated in the case of *The Midland Railway and Others v. Messrs. Butler and Co. (Bristol), Ltd., and Messrs. Brotherton and Co., Ltd.*, in which the Railway and Canal Commissioners held that they could not alter the *bona fide* judgment of the railway company that certain goods were of a dangerous nature.

In addition to the new tribunal, the Rates Advisory Committee recommends that a body, to be called the Railway and Traders' Conference, should be set up to act as a Conciliation Board without coercive powers. The proposal is that in each of the great distributing centres of the country two panels should be set up, one consisting of representative traders, and the other of officials of the railway companies serving the district; and that from these panels a committee should be formed to which any

traders having a grievance might make application, and which would have power to make recommendations but not to make or enforce orders. If a question appeared to involve general principles or an issue of great importance, the local tribunal would be empowered to refer it to the Rates Fixing Tribunal. The object of these local tribunals is to secure a hearing for traders who, although admitting that the railway authorities will usually redress a grievance if it be properly presented to them, nevertheless contend that their complaints are too often disposed of in a rough and ready manner by minor officials without adequate consideration.

**Maximum and Actual Rates.**—The maximum charges for the conveyance of merchandise which the railway companies are entitled to make under the Rates and Charges Order Confirmation Acts, 1891-92, were really a necessity so long as the railways were allowed to determine the actual rate, but as the power to fix rates is in future to be in the hands of the new tribunal, the Rates Advisory Committee suggests that it should also be entrusted with the determination of maximum rates. This appears very reasonable, seeing that the new tribunal is to have the power of raising or lowering rates after full investigation.

**Continuous Mileage.**—Throughout the evidence given by the traders, a general desire was expressed that the new system of fixing rates should be on the principle of continuous mileage, and not upon the cumulative principle now in operation. The effect of the cumulative principle is that rates for a distance of, say, 100 miles, when computed over two lines of railway, are greater than those for 100 miles when the transit is on one line of railway only; and the Rates Advisory Committee considers this state of things to be a serious anomaly. The railway companies strongly opposed the principle of continuous mileage, although actually many of the rates which they charge at the present time are based upon it, as are also, generally speaking, the receipts for the conveyance of merchandise when divided between the various railways, no regard being paid to any advantage that may have accrued where the rates are made upon the cumulative principle. The Rates Advisory Committee therefore recommends that in the case of through rates the principle of continuous mileage be adopted with provisions in the case of different rates prevailing on the railways involved similar to those in regard to portions of the railway having different maxima under the Railway Rates and Charges Orders.

One of the most important features of the recommendation relating to continuous mileage is its application to short distance traffic and to the fixing of the minimum charge. The report states:—"We consider the anomaly of making two minimum charges as for six miles each of transit, for possibly a total distance of four or five miles, as more objectionable than that of different charges for long-distance hauls according to whether the transit is on one or more railways." With the adoption of continuous mileage, the Rates Advisory Committee does not intend to interfere with the existing regulations, but to allow bonus mileage to be charged in respect of the portions of the lines which have been constructed at extraordinary expense, such as Runcoorn Bridge, Forth Bridge, Severn Tunnel and lines in and around London. It is intended that such bonuses should continue to be charged, and in general indicated by the total distance being treated as being increased by the stipulated extra mileage. In those cases where different maxima have been fixed in respect of certain portions of the same railway, the Committee thinks that these differences should not be continued, but that a single rate sufficient to provide the desired revenue should be fixed for each class of

goods upon the whole or each of the railways or groups.

**Terminals and Disintegration of Rates.**—Section 33, Sub-section 3, of the Railway and Canal Traffic Act, 1888, placed an obligation upon the railway companies to disintegrate rates when application is made by a trader, but the disintegrations which have been given by the companies in the past have been in such a form as to be practically useless to the trader asking for the information; and during the hearing the traders consistently urged that all rates in future should be disintegrated in such a manner as to show separately the difference between the amounts for conveyance and those for each of the services.

The railway companies are willing to concede that if standard rates and tariff are fixed by an independent authority, the rates and tariff so fixed should show the amounts to be charged for conveyance and for terminal accommodation and services separately, but they claim that there should be no obligation upon them to disintegrate exceptional rates or apportion any part of the charge to terminals. The Committee is of the opinion that there is "to some extent justice in the claims of the traders, but that the counter arguments of the railway companies ought not to be ignored," and it recommends that the conveyance rate and terminal charges should be separately specified in the standard rates to be fixed by the tribunal, and that when the tribunal is called upon to fix an exceptional rate it should specify how much of that rate is for conveyance and how much for each terminal.

In the main, the claims urged by the traders have been met in the recommendations of the Committee, and these will prove a great boon, especially to those traders whose works are connected with the railways by private sidings.

**Services Rendered at or in connexion with Private Sidings.**—The present position as regards private sidings is that the owner pays the conveyance rate, but is free from any general obligation to pay the terminal charges whether for station accommodation or services, but the Rates and Charges Order Confirmation Acts, 1891-92, provide that the railway companies may charge a reasonable sum for services rendered at or in connexion with traffic at sidings not belonging to the company, any difference arising under Section 5 of the Orders quoted to be determinable by an arbitrator appointed by the Board of Trade. In practice, however, the railways have made their charge for siding services the amount of the terminal charges that are included in the rate applicable to the nearest goods station. This may be the full statutory terminal charges or less, according to the amount of the rate charged.

Whilst the Rates Advisory Committee does not see any reason for altering this state of things, it recommends that the duty of determining any difference should be placed upon the new tribunal. With a view, however, to fixing the reasonable sum to be paid by the owner of a private siding during any period which may elapse before the question can be determined, either by an agreement or by a new tribunal, the Committee has framed a series of rules.

Other matters dealt with in the report are "Conditions Affecting Carriage by Rail," and the "Conveyance of Traffic at the Actual Gross Weight" (except in the case of timber).

The report, as a whole, is a very valuable document, and opens the way for a scheme whereby the railway rates of the future may be made very much simpler and therefore more helpful to all concerned. The traders have gained valuable concessions, which will doubtless be more appreciated as their effects come to be experienced.

## CORRESPONDENCE.

### RAIL CONVEYANCE OF CHEMICAL COMMODITIES.

SIR,—Mr. Lukes' article in the *Journal* of December 31 does not, I think, need a lengthy reply from me, and I would ask those who are interested in the subject to re-read my article in the *Journal* of November 15, and they can judge whether I succeeded in correcting the exaggerations in Mr. Lukes' first article, and whether he has now brought forward any new facts of material importance.

When Mr. Lukes asserts that the railway companies have the power to decide what goods are dangerous and the sum to be charged for their conveyance "whether their views be right or wrong," he exaggerates the facts. The law provides that the companies shall not exercise their powers unjustly, and their charges must be reasonable.

The quotation from the American Inter-State Commerce Commission Regulations appears to insinuate that the British railway companies have not conferred with manufacturers and shippers in regard to specifications and regulations for the packing and transport of dangerous goods, but Mr. Lukes knows that many such conferences have been held and amendments made as the result of such discussions. I do not know whether these conferences are compulsory in America, but I do know that the British railways are not compelled to hold such conferences, but have done so *voluntarily*.

In his reference to the test case heard before the Railway and Canal Commissioners, in the matter of the *North-Eastern Railway Co. v. Reckitt and Sons, Ltd.*, Mr. Lukes omits to mention that the presiding judge, Mr. Justice Banks, stated in delivering his judgment:—"In my opinion, therefore, even if the test contended for by the defendants be applied, these goods are dangerous." Mr. Gathorne Hurdy, although in some doubt, said:—"On the whole, especially having regard to the question of storing and warehousing, I cannot bring my mind to the conclusion that these are not dangerous goods." Sir James Woodhouse, although he held a different opinion, as stated by Mr. Lukes, made some very pertinent remarks which Mr. Lukes omits from his quotation. Sir James said, in reference to Section 105 of the Railway Clauses Act, 1845, and the companies' powers:—"They must of course exercise the discretion vested in them with absolutely good faith and not for the indirect purpose of obtaining charges which they could not otherwise obtain. There is no suggestion of their acting otherwise in this case." (The italics are mine.) Mr. Lukes complains of the arbitrary powers given to the railway companies, but he has not given a single instance in which the Court has decided that these powers have been used by the companies in an unfair and arbitrary manner.

I stated in my article that the rates charged for dangerous goods had not been generally raised as a consequence of their transference from the White to the Yellow pages. Mr. Lukes gives no instance to the contrary. He mentions trinitrotoluol; but this substance always has been classified as a dangerous article, and the change made has been to transfer it from the category of Dangerous Chemicals to the list of Explosives, consequent upon the issue of an Order-in-Council, No. 898 of 1917, which definitely included it in the Authorised List. The rates charged for its conveyance automatically became the same as those charged for all other explosives of the same class.

As regards "Spirits of Tar" and "Mineral Tar Oil," no rates were raised. In these cases, the traders claimed to declare their traffics under names which did not correctly describe them, in order to

obtain the benefit of lower rates, but the Court decided that these claims could not be upheld.

Private-siding rates and the contention that all railway rates should be based upon the actual cost and the capital actually employed in handling and conveyance of the particular traffic involved have been dealt with in the report of the Rates Advisory Committee recently issued by the Ministry of Transport, and no further word need be said here. This report also deals with the classification of dangerous goods, the railway companies' liability in connexion with them, the rates to be charged, and the indemnity against consequences. One clear statement in the report may be quoted which has been urged over and over again but never accepted by some traders or their representatives. It occurs on p. 10, and is as follows:—"But as soon as it is established that rates are from time to time to be so fixed that the railway companies (subject to the obligation of providing good management) are secure of receiving adequate net revenue, they have a much smaller concern in the question of lowering particular rates or classes of rates, being satisfied that the aggregate will be made good to them from some other source, while the traders generally have become deeply interested parties to the discussion, seeing that for the first time wherever a diminution of revenue is caused by concessions to one class it will have to be made good by throwing the burden to the rest of their body. In other words, whenever a proposal is made in the general revision of rates for favourable treatment for any class of commodities the general body of traders have a greater interest in opposing it than have the railway companies. Indeed, but for the fact that the knowledge and experience of the railway companies' officials are necessary to secure a scheme that will work smoothly and efficiently, those companies might stand on one side and say: 'Let the traders decide among themselves how they desire that the necessary revenue should be raised.'" May we hope that now that this truth has been pronounced by the "impartial business tribunal" appointed by the Ministry, on which there was only one railway representative, after an exhaustive inquiry, it will be accepted, and that the agitation founded on other ideas will cease?

In concluding these remarks, I again acknowledge with thanks the valuable assistance of some of my railway colleagues.—I am, Sir, etc.,

L. ARCHBUTT.

## MEETINGS OF OTHER SOCIETIES.

### ROYAL SOCIETY OF ARTS.

Three Cantor Lectures on "Micro-organisms and some of their Industrial Uses" were delivered on November 29, December 13 and 20, by Mr. A. Chaston Chapman, Dr. M. O. Forster presiding.

Commencing with the discovery of zymase by Büchner, the lecturer briefly described Harden's work on the enzymic nature of fermentation, showing that the presence of a co-enzyme was as necessary as that of the zymase itself. The mechanism of alcoholic fermentation was then dealt with at some length, and it was shown that pyruvic acid and acetaldehyde were intermediate products formed during the fermentation of sugar under certain conditions.

The importance, from the point of view of chemical industry, of being able to modify cell-functions by varying their environmental conditions was indicated and illustrated by several examples. The production of glycerin on a large scale during the war by the fermentation of sugar

was described and the results obtained in Germany and in the United States were referred to.

The Amylo process for the production of alcohol on the large scale was next dealt with somewhat fully, and the nature of the improvements in the process made during recent years and the directions in which further improvements might be looked for were indicated. It was pointed out that about 20 million hectolitres of absolute alcohol had been made on the Continent by this process since its introduction, twenty years ago, but that, owing to excise restrictions, it could not be employed in this country. The use of bacteria for liquefying starch in the Amylo process for the purpose of reducing the waste of nitrogen was also described.

Further illustrations of the technical application of microbiology to industrial chemistry were instanced in the manufacture of citric, pyruvic, and fumaric acids by so-called "fermentation" processes. The manufacture of lactic acid was somewhat fully described, and the lecturer pointed out the unprogressive character of the industry in this country, the technical conditions to-day being but little better than at the commencement. The manufacture of butyric acid was stated to be in an even worse position, as the best and most economical conditions for the biochemical production of this acid were far from being known. The manufacture of vinegar was next described, and it was pointed out that, although the industry is worked on sound lines—so far as the preparation of the alcoholic wash is concerned—it is on a much less satisfactory basis on the biological side. The working losses experienced at present are often very considerable, and most manufacturers have little or no knowledge of the actual biological character of the organism or organisms they are using.

At the commencement of the third lecture the biochemical production of butyl alcohol and acetone from starchy materials was dealt with, and reference was made to the results obtained during the war at King's Lynn, at H.M. Naval Cordite Factory at Holton Heath, and at the works of the British Acetone Co. at Toronto (cf. J., 1919, 271 r, 273 r, 282 r, 411 r). The mechanism of the process is very complex, and in order that it might compete with the ordinary process for the production of acetone, cheap raw material must be available, as well as a market for the butyl alcohol, the hydrogen formed and the constituents of the spent wash. The conversion of butyl alcohol into methylethylketone was a possible solution of one of the above problems.

The lecturer then referred to the use of certain of the lower fungi for the production of nitrogenous foodstuffs. Brewers' yeast constitutes a valuable cattle food or, when de-bittered, can also be used as a constituent of manufactured food products intended for human consumption. The use of yeast for the manufacture of an extract closely resembling extract of meat in its chemical and physical properties was referred to, and it was mentioned that a well-known brand of yeast-extract at present on the market had been found to be rich in the water-soluble vitamins or accessory growth substance, whilst meat extract had been found to be devoid of this important growth-promoting constituent.

The use, for food purposes, of the so-called "mineral yeast" in Germany during the war and its manufacture in this country were described (cf. J., 1919, 285 r). The discovery of a simple biochemical method for the conversion of cellulose into sugar was mentioned as affording a probable solution of the power alcohol problem and also of rendering possible the economical production of synthetic nitrogenous foodstuffs.

In conclusion, Mr. Chaston Chapman pointed out how backward England had been in connexion with important discoveries in the domain of industrial

microbiology, and renewed his plea for the foundation of a National Institute devoted to that subject (*cf. J.*, 1919, 282r). The function of such an institute would be to provide for the systematic prosecution of research in connexion with all those industries in which micro-organisms or enzymes play an important part. In addition to this it would serve as a centre for the specialised training of men intending to devote themselves to the teaching of microbiology and biochemistry in our universities and technical schools, as well as for the practical instruction of technical employees engaged in the various biochemical industries. It would also provide breweries, distilleries, and other factories with any required organisms in pure culture, and would serve generally as a central home for the promotion of microbiological science.

## NEWS AND NOTES.

### SOUTH AFRICA.

**Lead and Vanadium in the Transvaal.**—A deposit of lead and vanadium, probably of considerable extent, with a content of lead metal ranging from 8.15 to 15.5 per cent., and of vanadium pentoxide from a trace to 1.35 per cent., occurs at the old Doornhoek Lead Mine, Marico District.—(*S. Afr. J. Ind.*, Nov., 1920.)

**Maroola Nuts as a Source of Oil.**—A sample of Maroola nuts (*Sclerocarya caffra*—a tree that occurs extensively in the Transvaal), has been investigated at the Imperial Institute, which reports that the nuts consist of about 90 per cent. of shell (very tough and fibrous), and only about 10 per cent. of kernel. The kernels yielded 59.2 per cent. (dry basis) of a non-drying oil which would be suitable for soap manufacture and possibly for edible purposes. It is very improbable that the nuts could be used as a source of oil owing to the difficulty with which they are cracked and the kernels separated.—(*S. Afr. J. Ind.*, Nov., 1920.)

**Platinum in the Lydenburg District.**—Pending reliable information concerning the alleged important discovery of platinum in the Cala district, it may be recalled that this metal is known to occur in other parts of the Union. Pseudo-stratified segregations of chromite associated with platinum occur for considerable distances along the margin of the Bushveld granite area in the Transvaal. The segregations are several inches thick, and though of workable size and extent have never been exploited. The chromic-oxide content is usually between 35–45 per cent., but 54 per cent. in places, and the Mines Department of the Union states that the ores contain an appreciable quantity of platinum. Platinum and allied metals are also found in the black sands derived from the conglomerates in the Klerksdorp area, a district which is at present very inaccessible, but the occurrence is stated to be well worth attention.—(*Official.*)

**Sulphur in South Africa.**—The imports of sulphur into the Union of South Africa during 1913 and 1917 were as follows:—

	1913.	1917.
Sulphur (long tons) ...	2672	899
Pyrites (     "     ) ...	22,903	18,465
Sulphuric acid (lb.) ...	291,976	37,435

Five factories in the country produce sulphuric acid for their own use, and for trade when supplies of pyrites and railway facilities are available. The present demand for pyrites amounts to 1650 tons per month; shortly it may be 3500 tons. Deposits of native sulphur are known to occur in

the Union, but none of these, so far as is known, could be profitably worked. Railway facilities are needed for the development of the massive pyrites deposits occurring in the Areachap Copper Mine, 10 miles from the station. Disseminated pyrites is plentiful in the gold-bearing rocks and in the coal mines. It is estimated that during next year coking plants in the Union will supply about 450 tons of pyrites per month. Pyritic auriferous concentrates, derived from the Sandstone Reef, afforded a monthly supply of 400 tons of pyrites in 1919, and it is thought that supplies from this source can be increased to 1000 tons. The remaining requirements of the Union may possibly be met by the utilisation of pyrites derived from concentrates produced in the Barberton district; the presence of arsenic, to the extent of about 1 per cent., has hitherto hindered their exploitation.—(*S. Afr. J. Ind.*, Nov., 1920.)

### AUSTRALIA.

**Yacca Gum in South Australia.**—The collection and export of yacca gum was an important industry in Kangaroo Island, South Australia, prior to 1914, but operations were suspended during the early years of the war as the chief market, Germany, was closed. A revival, however, took place in 1916, and in 1919 over 10,000 tons was collected, most of which was exported to Great Britain and the United States. The gum is soluble in alcohol and caustic alkalis, but insoluble in turpentine, linseed oil, benzene, and hydrocarbon solvents generally; it is stated to be serviceable in the manufacture of photographic chemicals, dyestuffs, and linoleum (*cf. J.*, 1918, 392 r).—(*U.S. Com. Rep.*, Nov. 19, 1920.)

### NEW ZEALAND.

**Mining and Minerals in 1919.**—The Mines Statement for 1919, by the New Zealand Minister of Mines records that legislation will shortly be introduced to revive the bonus provisions of the Iron and Steel Industries Act, 1914, and that arrangements are being made to have 20 tons of iron sand from Taranaki and 20 tons of iron ore from Parapara tested in England by a new process in order to determine if it is possible to produce iron on a commercial scale in New Zealand. Since September, 1919, the Government has been negotiating with the British Admiralty with a view to making an arrangement with the British Government for boring and developing mineral oil and other products in New Zealand, but no agreement has yet been made. Unfortunately, petroleum has yet not been found in payable quantity in New Zealand, but the Government is now taking steps to settle definitely whether the oil resources are of commercial value.

During the year 1919 gold to the value of £573,662 was obtained from quartz mines, 286,073 tons of ore being treated; the value from alluvial and dredge mining was £128,111. There was practically no demand for tungsten ore (scheelite) for the English market; the price fell from 68s. to 30s. per unit, and consequently the production was very small—131 tons, compared with 169.5 t. in 1918. The mining of cinnabar and production of mercury were carried out by one company, and 11,175 lb. of metal was produced. The Mines Department is assisting the company to test the lode on its property, and improved returns are looked for in the near future. The quantity of phosphate rock quarried was 4000 t., as against 5000 t. in 1918. The coal output was 1,847,848 t., compared with 2,034,250 t. in 1918, the decrease being due to organised restriction of work by the miners on several fields. The production of kauri gum rose from 2419 t., valued at £157,313, in 1918, to 4128 t., worth £255,812, in 1919. A company with a capital of £200,000 has been formed to produce oil and gum from kauri peat, of which it possesses some



6½ million cb. yds. on its properties. As the yield is estimated to be £1's worth of fine gum per cb. yd. of peat, a large production is anticipated.

#### UNITED STATES.

**Coloured Plasters.**—The production of coloured plasters has been made possible by simply mixing dyed wood fibres with gypsum. A series of samples has been made and tested by the U.S. Bureau of Standards.

**Charcoal from Walnut Shells.**—Experiments made in California appear to have overcome the difficulty attending the retorting or charring of dusty cellulosic materials such as walnut shells. The ground shells are passed through the hot zone of a flame which completely burns off the dust, baffles and lifters ensure adequate mixing, tar is recovered from the exit gases, and the resulting charcoal is quenched as it is discharged from the kiln. The walnut-shell charcoal is used instead of willow charcoal for poultry.

**Use of Cyanamide and Superphosphate Mixtures as Fertiliser.**—The toxic effects on plant life due to the application of cyanamide mixed with superphosphate are now reported to be caused by the rapid formation of dicyanodiamide. Conversion of cyanamide into dicyanodiamide by moisture is much accelerated in the presence of superphosphate; calcined and basic phosphates exert no action. The use of a dry mixture is of little avail as the soil moisture may bring about the change under certain conditions.

**Lime in 1918.**—The total quantity of hydrated lime produced in 1918 was 620,216 short tons, valued at \$5,342,513, and the total quantity of lime sold amounted to 3,206,016 short tons valued at \$26,808,909, an average price of \$8.36 per ton. The building trade consumed 28.5 per cent., chemical works absorbed 17.7, and agriculture used 12.2 per cent. of the total production. Imports amounted to 6650 t. and exports to 7191 t.

At least five companies used lignite as fuel, one producer reporting that it compared favourably with other fuels, both in regard to cost and the quality of lime produced. A lignite containing a large percentage of moisture produces a soft-burned lime similar to lime burned with wood. It usually requires about half a ton of lignite to burn a ton of lime. To be used efficiently the kiln should have a fire-box with large grate area and a high arch or combustion chamber; it should also have a taller shaft than usual since the gases in lignite distil off very rapidly and would otherwise burn off on the top of the kiln. After storing lignite a week or two, it will produce only about half the quantity of lime it does when fresh.—(*U.S. Geol. Surv.*, June 7, 1920.)

#### JAPAN.

**Opium Production.**—The production of opium in Japan has made very great strides during the past few years, as the following official statistics show:—1913, 2196 cwt.; 1914, 2877 cwt.; 1915, 7516 cwt.; 1916, 22,774 cwt.; 1917, 28,792 cwt.; 1918, 10,123 cwt.; 1919, 33,731.5 cwt. During 1919 the poppy was cultivated in thirty Prefectures of which Osaka, Wakamaya, Hojogo, Yamayachi, and Seitana were the chief. The average morphine content of the 1919 crop was 12.68 per cent., which was 3 per cent. lower than that of the crop in 1918.—(*Chem. Ind.*, Dec. 15, 1920.)

**The Cement Industry.**—The Japanese cement industry is in a flourishing condition; the formation of several new companies is announced, and most of the existing companies have extended their activities. The combined paid-up capital of twelve old-established firms increased from £3,200,000 in 1914 to £4,460,000 in 1919, and the price of cement rose from 6s. 1½d. per 400 lb. to more than four

times that amount during the same period; similarly, the production increased from 3,625,000 to 6,500,000 barrels, and the exports from 258,000 to 997,000 barrels.—(*Bull. Dept. Tr. and Com.*, Nov. 22, 1920.)

**"Koka Seki" Pumice.**—"Koka seki" is a variety of pumice stone found only in the Niiijima Islands, about 90 miles south of Tokyo. It has long been used as a building material, and on account of its durability, high tensile strength, and resistance to heat (1300° C.), it can be used in boilers and furnaces as well as for lining safes and refrigerators. Its most important use is in the construction of reinforced concrete barges. The concrete is stated to be about 60 per cent. lighter than the ordinary kind, and to be absolutely resistant to seepage, erosion, etc. "Koka seki" has recently been selling in Japan at about 2s. per cb. ft. for blocks and 8d. per cb. ft. for flakes and sand.—(*U. S. Com. Rep.*, Oct. 26, 1920.)

#### FRANCE.

**Industrial Notes.**—*Coal.*—The high costs of coal and labour, together with shortened hours of work are responsible for the continuance of the industrial crisis. The economic situation has been seriously affected by the excess of supply over demand, imports having exceeded exports and Allied war stocks having been released. The high cost of coal in particular is a serious handicap, for the price is in France very much higher than in any of the neighbouring countries, and manufacturers are therefore unable to cope with foreign competition. The remedy suggested is not a high tariff wall, but compensatory duties to equalise the production costs to competitors. The decontrol of the coal industry is being considered by the Minister of Public Works and the Coal Commission has been instructed to prepare a scheme to ensure supplies until decontrol is complete. The output of coal in October was 2,250,880 tons, and of lignite 85,585 t., whilst 846,629 t. was produced in the Saar basin. Germany exported 1,814,884 t. during November, of which 1,242,974 t. went to France. Imports of American coal continue to increase; in January, 1920, 20,000 t. was imported at \$30-32 per ton, and in November, 1920, 700,000 t. at \$15-16 per ton.

**Chemical Industry.**—The formation of large chemical trusts in the United States, in Great Britain, and, to some extent, in France, is being criticised on the ground that such combinations tend to develop bureaucratic methods and thus lead to inefficiency. The critics would prefer a system of agreements between producers under which each would specialise in certain products and supply prescribed areas. Such a system, they say, leaves to each partner his own responsibilities and interests, but allows him to reap the advantages accruing from concerted action.

Great progress is being made by the Société des Matières Colorantes et Produits Chimiques de St. Denis, which extended its works in 1919 with a view to producing all the raw materials and intermediates required in dyestuff manufacture. In June, 1920, it was producing coal-tar intermediates and dyes at the rate of 135 and 150 tons a month, respectively, and as soon as certain extensions are completed, these figures will be more than trebled, thus allowing an ample margin for export.

The Société de la Grande Paroisse intends to erect at Montereau a synthetic nitrogen plant (Claude process) with a daily capacity of five tons of liquid ammonia, and the Société Norvégienne de l'Azote is to extend its nitrogen works at Soufom.

**Metallurgy.**—The demand for metallurgical products is very small in view of the expected decline in prices. It is unlikely, however, that prices will fall to any extent until the large stocks



of foreign coal bought at high prices have been liquidated. The reported agreement between French, Belgian, and Luxembourg producers of cast iron with a view to stabilising prices has not been ratified.

#### GENERAL.

**Chaulmoogra Oil.**—In a recent number of the *Journal of Tropical Medicine and Hygiene*, Dr. T. A. Henry, Director of the Wellcome Chemical Research Laboratories, describes some recent developments in the use of chaulmoogra oil in the treatment of leprosy. It appears that Dr. J. McDonald and Professor Dean, working in Hawaii, have obtained promising results by the treatment of this disease with the ethyl esters of the fatty acids peculiar to chaulmoogra oil. These acids were thoroughly investigated by Dr. F. B. Power and his collaborators some years ago and shown to differ from ordinary fatty acids in constitution and in being optically active.

Sir L. Rogers and others working in India have improved on the old method of administering chaulmoogra oil itself by using the sodium salts of fractions of the fatty acids for injection and also for oral administration, and the method in use in Hawaii appears to be a further development of this, by the use of fractionated ethyl esters in place of the sodium salts. Mechanically, at all events, the esters are better than the salts for injection. Rogers and his collaborators have recently found that they can get equally good results with the sodium salts of fatty acids from other oils than chaulmoogra, e.g., gynoecardia oil, and it therefore becomes a question whether the efficacy of certain oils as remedies for leprosy, and possibly also for tuberculosis, is not common to many oils and due, as has already been suggested, to their solvent power on the waxy external coating of acid-fast bacilli. Against this view is the evidence brought forward by Walker and Sweeney in California that the action of the chaulmoogra-oil fatty acids is specific against the leprosy bacillus *in vitro*.

**Agricultural Industries in Jamaica.**—According to a recent report of the Department of Agriculture, Jamaica, the period June, 1919, to June, 1920, marked the greatest industrial revival which the island has experienced during the past century. The recorded exports for 1919 increased by over 100 per cent. in value over those of the previous year, and there appears to be every prospect of stable markets for all the chief products of the various industries. Although Jamaica now produces many goods that were formerly imported, much still remains to be done, and it has been suggested that the following industries, *inter alia*, could be profitably initiated:—Condensed milk, cement, glass bottles, rope and string, and leather.

Marked progress was made in the sugar industry, exports being 38,000 tons, valued at £1,333,000, and £225,000 inclusive of rum. This record is expected to be much exceeded in 1920. The banana industry made a welcome recovery in the absence of hurricanes, and there were exported 10 million bunches of bananas and 20,000 boxes of evaporated bananas. The coconut crop showed a progressive recovery from the effects of the cyclone in 1917, with an export of over 24 million nuts and nearly 700 tons of copra. At no period of its history has Jamaica been so free from coconut diseases as at the present time, and the high value of the products is an incentive to increased cultivation. Logwood and its extracts have, contrary to expectation, enjoyed a revival of prosperity under post-war conditions, and an improvement of £60,000 over last year's values was recorded. Logwood is in strong demand at high prices, and all the local factories are working at full capacity. Orange oil realised good prices, and crop values are nearly double those of the previous year. Cacao had a

good year, with an output value 90 per cent. greater than in 1919. Cotton cultivation has received some attention, and a small planting of Sea Island cotton resulted in a handsome profit to the grower. Pimento was in good demand abroad, the exports being valued at £184,000. Experiments on the production of pimento oil and its commercial conversion into vanillin are now being carried out at the Government Laboratory; if these are successful, Jamaica should command the world's markets for vanillin.

The production of sisal fibre is becoming an important new industry. The Government has already embarked on a large sisal enterprise at Lititz, where it has been shown that a minimum output of 1000 lb. of fibre per acre in the first cutting can be relied upon. This fibre has been thoroughly investigated in London, and offers have been received to buy all that can be produced at a very remunerative price. It is estimated that fibre to the value of £100,000 could be produced annually on land which has hitherto been comparatively unproductive. Hides were in good demand. When the tick question has been solved by the compulsory dipping regulations which will shortly come into force, a decided appreciation in the value of the hides produced in Jamaica should result.

**Pimento-leaf Oil in Jamaica.**—Investigations at the Government Laboratory of Jamaica have shown that the pimento leaf yields about 1·8 per cent. of eugenol, from which iso-eugenol and vanillin can be obtained. Iso-eugenol can also be obtained by the appropriate fermentation of pimento leaves. Eugenol could be used in the manufacture of vanillin and, as it is a strong antiseptic, it would be a suitable constituent of dental and toilet preparations. Jamaica could produce 100,000 lb. of pimento-leaf oil per annum from waste materials were a market available.—(*U.S. Com. Rep.*, Nov. 1, 1920.)

**Borates (1913—1919).** (*Imperial Mineral Resources Bureau*, Pp. 24. Price 9d.).—The natural borates of industrial value include, in the order of importance, (1) ulexite (hydrated borate of sodium and calcium), (2) colemanite (hydrated calcium borate), (3) hornblende or Turkish pandermite (borate and chloride of magnesium), (4) sassolite (native boric acid occurring chiefly in solution), and (5) borax (hydrated sodium borate).

As is well known, borax is used for many purposes, e.g., as a flux in brazing and brass manufacture, as a constituent of pottery glazes and enamels, and as a glaze for paper and linen. It is employed in the tanning, glue, glass, and soap industries, and the borates of manganese and lead are used as drying agents in the paint trade. The perborates of calcium and sodium find application as a source of pure hydrogen peroxide, and hence also in the production of some modern bleaching powders.

Boron in the form of a suboxide is used to remove oxygen from copper just before casting, the conductivity and density of the metal being thereby greatly improved. It is also used in the manufacture of strong aluminium bronzes, in hardening and strengthening aluminium castings, and in the manufacture of nickel castings. Experiments conducted in France during the war indicated that remarkably strong and tough steels can be produced with the aid of ferro-boron. Another use of boron is as a voltage regulator in incandescent lamps, arc lamps, and other devices on series circuits.

The United States is now the great producer of crude borates, the production during the last three years amounting to over 80,000 metric tons per year. No analysis of the crude material is given, so that it is impossible to gain any idea as to the relative importance of the producing countries in terms of refined borax. The deposits are situated

throughout the Great Basin region, and in particular in the counties of Inyo, Kern, and San Bernardino in California and in the adjacent part of south-west Nevada. The borates are found in marshes and dried-up lakes, and are of a secondary character, being derived from beds of colemanite in the Tertiary lake sediments of the surrounding hills. Chile, Peru, and Bolivia rank next in order of importance as producers of borates. The western range of the Andes of South America contains many lofty volcanoes recently extinct, and around the base of these mountains there exist borax lakes sufficiently numerous to supply the world for many centuries to come, although but few of them have yet been developed. All of these deposits are owned and worked by a British company, Borax Consolidated, Ltd. No part of the British Empire produces borates at the present time. Our imports of calcium borate, borax, and boracite during 1919 amounted to 325,826 cwt., valued at £387,169, of which 13,416 cwt., worth £25,845, was exported.

The report contains detailed statistics concerning the foreign trade in borax and boric acid of the various portions of the British Empire, and interesting notes on the character of the world's deposits. It is to be regretted, however, that no statistics are given showing the production of refined borax in the chief producing countries.

**The Petroleum Industry of Mexico.**—Although the United States contributes about two-thirds of the oil supply of the world, 40 per cent. of the producing fields have become exhausted, and it is estimated that the oil reserves will be depleted within the next twenty-five years. The United States is at present an importer of oil, and this condition is bound to continue and to become more and more apparent. Mexico, ranking second as an oil producer, offers scope and encouragement not only to the American oil interests, but also to the Eastern hemisphere. Mexican production has increased threefold in the last seven years, as the following table shows:—

Year.	Mexico.	U.S.	World.
1913	25,902,439	248,446,230	384,667,550
1914	21,188,427	265,762,535	399,667,168
1915	32,910,508	281,104,104	426,370,894
1916	39,817,402	300,767,158	459,433,319
1917	55,292,770	335,315,303	505,362,367
1918	63,828,327	355,927,716	614,729,354
1919	87,359,533	377,719,000	

(The production is given in barrels of 42 galls. capacity)

**Potential and Actual Production.**—The potential production, i.e., the amount that could be produced were each well allowed to flow without restraint, of Mexico during 1918 was 547 million barrels. Only about 12 per cent. of the potential capacity of the wells is being actually produced at the moment. This is partly due to lack of transport both for oil and materials, partly to lack of storage facilities, and partly to the political situation; moreover, new legislation has tended to check development.

**Comparison of Mexican and American Wells.**—The oldest wells in America are in the Appalachian field, and number about 100,000. The average production is  $\frac{1}{4}$  barrel per well per day; in the Californian field the average is 30 hrs. daily per well, whilst there are 25 wells in Mexico which could produce freely 24,000 hrs. per day per well.

**New Mexican Fields.**—The zone of potential production in the Gulf Coast States is estimated at 80 million acres, in the Pacific Coast at 50 million, and in Lower California at 18 million acres, a total, in all, of 230,000 square miles—of which only 6½ million acres have been investigated. The actual producing fields cover 800 sq. miles. Geologists predict that a paraffin base oil may be looked for,

or, at any rate, an oil of lower asphalt content than that produced at present.

**The Salt Water Menace.**—The Terpetate, Huasteca and Casiano fields have been seriously invaded by salt water. In November, 1919, the Casiano 7, producing 25,000 barrels per day, turned to salt water. This phenomenon will probably stimulate the exploration of the southern fields.

**Pipe Lines and Transport.**—Production is greatly restricted by insufficient transport. Pipe lines of total carrying capacity of 300,000 barrels per day are under construction and projected. Some of the very heavy and viscous oils cannot be moved in lines, tank cars are necessary, and even then heating facilities must be provided. The Tampico Panuco railroad is the most important common carrier in the oil region.

**Storage and Refineries.**—A great surplus of crude petroleum is stored in earthen reservoirs, awaiting improvement in transport facilities. In January, 1919, the tankage was 49 million hrs., mainly steel, but to a limited extent concrete. There is a great need for refineries, and the Mexican Government proposes to encourage their establishment by the grant of concessions and franchise. There are four plants at Tampico, one each in Tuxpam, Vera Cruz and Minatitlan, with a total capacity of 100,000 hrs. per day. About 20 per cent. of the oil exported is refined. New refineries are being contemplated and erected at Panuco, Tecomaite, Tampico, and Mexico City.

**Statistics for 1920.**—For the first six months of the current year the oil exports from Mexico reached 60 million hrs., a 72 per cent. increase over the corresponding period of 1919; the United States took 71 per cent. of the total production, South America 8 per cent. and Great Britain 4 per cent. The consumption of oil in Mexico itself amounts to 4 per cent. of the production. —(Report of the Bureau of Foreign and Domestic Commerce, U.S.A.)

**Sesame Seed in Mexico.**—The production of sesame seed in Mexico is capable of great expansion, as the climate of the chief producing states, Michoacan, Guerrero, Morelos, and Vera Cruz, is admirably suited to the crop, and large areas of untilled land are available. The white and brown varieties of sesame grown in the country have an oil content officially estimated at 50 per cent., but although the former is stated to be richer in oil the latter grows more rapidly and produces more crops in a given period of time. The methods employed for separating the oil are said to be more modern than those used for olive oil in Spain and Italy. The first-grade oil is almost odourless and is used as a substitute for olive oil as well as in the manufacture of oleomargarine and in the adulteration of other edible oils. The inferior grades, which are usually rancid, are used for soapmaking, as a combustible, and as a basis for perfumes. The cake remaining after extraction is similar to cottonseed cake in value and is used as a cattle food. The leaves of the plant have medicinal properties. —(U.S. Com. Rep., Oct. 30, 1920.)

**The Asbestos Industry in Russia.**—The Russian asbestos industry suffered greatly during the war, and the production fell from about 24,500 long tons in 1913 to 4000 t. in 1919. The industry is situated in the Urals, and prior to the war 70 per cent. of the production was taken by Great Britain, Germany and Austria. Great difficulty is being encountered in the work, especially with labour, owing chiefly to the seasonal character of the operations. The labour costs for 1920 are estimated at over 52 million roubles, a much higher sum than in previous years and more than double the total capital invested in the industry. —(Gummi-Zeitung, Oct. 29, 1920.)

**The Belgian Iron and Steel Industry.**—The present position of the Belgian iron and steel industry, as compared with that in 1913, is shown by the following statistics:—On October 1, 1913, 50 out of 58 blast furnaces were alight and the daily production of all pig iron was 7272 tons; on the same date in 1920, 21 out of 51 blast furnaces were alight, 30 being under reconstruction or not in use, and the daily production of pig iron was 3354 tons. Since July 1, 1920, the average daily production of pig iron for steel manufacture has increased by 22 per cent., that of pig iron for castings has increased by 100 per cent., and four more blast furnaces have been blown in.—(*Bd. of Trade J.*, Dec. 2, 1920.)

**Lectures on Glass.**—The British Lampblown Scientific Glassware Manufacturers' Association has arranged a series of lectures in connexion with the Lampblown Glass Industry, to be held at the Northampton Polytechnic Institute, Clerkenwell, E.C. 1, on Wednesdays at 7.30 p.m. from January 5 to March 23, inclusive. Prof. W. E. S. Turner will lecture on Glass Tubing (Jan. 26); Mr. English on Glass Manipulation at the Bench (Jan. 19); Mechanical Devices (Feb. 2), Graduation of Apparatus (Feb. 9); Mr. Higgins on Thermometry and Thermometers (Feb. 16, 23, Mar. 2); Mr. Stott on Volumetric Glassware (Mar. 9, 16), and Mr. Davis on Chemical Glassware (Mar. 23). The fee for non-members of the Association is 10s. 6d. for the course, or 2s. 6d. per lecture. Further information may be obtained from the Secretary of the Association, Mr. H. W. Ashfield, at 2/3, Duke Street, St. James, S.W. 1.

## PERSONALIA.

The honour of knighthood has been conferred upon Prof. James Walker, professor of chemistry in the University of Edinburgh.

Dr. Harriette Chick has been awarded the Mickle Fellowship of the University of London for her work on diseases due to defective nutrition.

Mr. A. W. Ambrose has succeeded Mr. J. O. Lewis as chief petroleum technologist to the United States Bureau of Mines.

Dr. C. J. Witt, assistant professor of analytical chemistry in the University of Pittsburgh, has become chief research chemist to the Portland Cement Association, U.S.A.

Dr. C. O. Johns, head of the colour laboratory of the Bureau of Chemistry, U.S. Department of Agriculture, has been appointed director of a new department of general research of the Standard Oil Company, New Jersey.

Dr. W. H. Walker has resigned from the directorship of the Division of Industrial Co-operation and Research of the Massachusetts Institute of Technology, and Mr. C. L. Norton, professor of industrial physics at the Institute, has been appointed in his stead.

Prof. Fr. Dolezalek, founder and director of the Physical and Electrochemical Institute in the Technical "Hochschule" of Berlin, died on December 10, aged forty-eight.

We regret to announce the death on December 1 last of Mr. Robert Tervet, of Leyton, Essex, an original member of this Society, who contributed papers on destructive distillation and on the production of ammonia from coke, showing the possibility of obtaining a yield of ammonia equivalent to 57 lb. of sulphate of ammonia per ton of coal by passing hydrogen over the incandescent coke remaining after carbonisation, the hydrogen combining with the residual nitrogen of the coke.

## PARLIAMENTARY NEWS.

### HOUSE OF LORDS.

In the House of Lords on December 23, Lord Cawley gave notice of his intention to move next session for a Committee of Inquiry into the circumstances attending the formation of British Dyestuffs Corporation, Ltd., and into its connexion with the Government.

### HOUSE OF COMMONS.

#### Exports of Cement.

Replying to Lieut.-Col. Croft, Sir R. Horne said that there was now a shortage of cement in this country, but the output was improving. The exports for the first eleven months of 1920 represented about 23 per cent. of the output, which was, roughly, the pre-war proportion, and consisted of 246,184 tons (£1,338,000) sent to foreign countries and 318,939 t. (£2,022,000) sent to British Possessions.—(Dec. 20.)

#### Substitutes for Salvarsan and Neo-Salvarsan.

In answer to Mr. Waterson, Dr. Addison said that the following substitutes for salvarsan and neo-salvarsan had been approved:—Kharsivan, neo-kharsivan, arsenobillon, novarsenobillon, diarsenol, neo-diarsenol, galyl, and salvarsan (manufactured by an English firm). These drugs were all officially tested for toxicity before being placed on sale, and there was good reason to believe that, with the exception of galyl, they all corresponded in general chemical constitution to the analogous German preparations.—(Dec. 20.)

#### Metalliferous Mines (Legislation).

Mr. Bridgeman, answering Mr. Cape, said that a Metalliferous Mines Bill was being prepared, but it was improbable that it could be introduced next session.—(Dec. 21.)

#### Coal Output.

In a written answer to Mr. Purchase, Mr. Bridgeman stated that the coal output during the week ended November 20 (the first full week since the coal strike) and during the three following weeks was 5,210,700, 5,176,500, 5,176,200, and 5,205,400 tons respectively. Weekly figures for the corresponding pre-war period were not available. Until the figures for the last week of the test period were obtained it was impossible to state how the wages scale would be affected. The present position of the industry did not warrant any hopes of cheaper home coal.—(Dec. 21.)

#### Wet Carbonising, Ltd.

Mr. Bonar Law, in a written answer to Mr. Bottomley, stated that Wet Carbonising, Ltd., guaranteed a contract by its subsidiary, Lochar Ltd., for the purchase of the Ironhurst Peat Fuel Factory, near Dumfries, from the Government for £370,000. These companies had been unable to raise the necessary capital, and Wet Carbonising, Ltd., would be responsible to the Government for any deficit that might ensue if the factory were sold elsewhere for less than the guaranteed price.—(Dec. 22.)

The Royal Assent has been signified to the British Empire Exhibition (Guarantee) Act, 1920, to the Women and Young Persons (Employment in Lead Processes) Act, 1920, and to the Employment of Women, Young Persons and Children Act, 1920. The Electricity (Supply) (No. 2) Bill was withdrawn on December 20.

Parliament has been prorogued to February 15, 1921.

## GOVERNMENT ORDERS AND NOTICES.

**IMPORT AND EXPORT RESTRICTIONS, ETC.**—The importation of hops is prohibited, save under licence or for transshipment, as from December 28, 1920.

All licences for the export of whisky granted prior to January 1, 1921, will be revoked as from February 1 next.

The following articles have been removed from Lists A and B of prohibited exports, as from December 30, 1920:—Potash, muriate and sulphate, crude manurial potash salts, and mixtures containing any of these substances; potassium permanganate; sugar, beet and cane.

The Commissioners of Customs and Excise have issued alterations in the Import and Export List, and the revised List will be published shortly. The *Board of Trade Journal* for January 6 contains a revised list of the foreign countries and British Possessions for use in the Monthly and Annual Trade Returns.

**IMPORTATION OF DYES UNDER THE DYE-STUFFS (IMPORT REGULATION) ACT, 1920.**—The Board of Trade announces that applications for import licences required for any goods covered by the Act should be addressed to the Secretary, Dyestuffs Advisory Licensing Committee, Danlee Buildings, 53, Spring Gardens, Manchester. Applicants must supply full information concerning the wares they wish to import.

**APPOINTMENTS UNDER THE GAS REGULATION ACT, 1920.**—The Board of Trade has appointed Sir R. T. Glazebrook to be Chief Gas Examiner, and Messrs. O. V. Boys, J. S. Haldane, and W. J. A. Butterfield to be Gas Referees. Mr. H. C. Honey is appointed Director of Gas Administration in the Power Transport and Economic Department. Board of Trade Offices, Great George Street, S.W. 1. A committee consisting of Sir W. Pearce (chairman), Dr. T. Caruath, Mr. W. D. Gibb, and Dr. T. Gray, has been set up to inquire into the necessity or desirability of prescribing a limit to the carbon-monoxide content of gas used for domestic purposes; and also a committee, consisting of Mr. J. H. Gray (chairman), Mr. W. J. Butterfield, and Dr. C. H. Lander, with similar terms of reference, but relating to incombustible constituents.

**PROPOSED NEW REGULATIONS FOR CHEMICAL WORKS.**—The Home Secretary has issued a draft of the new regulations he proposes to make to supersede those made in 1908 for the manufacture of nitro and amido derivatives of benzene and those made in 1913 for the manufacture of chromate and bichromate of sodium and potassium. The new regulations are divided into those which are applicable to all chemical works, as defined in the schedule, and those relating to works in which there are special dangers. The manufactures of certain lead compounds, already dealt with in special codes, are excluded, and also processes of bleaching, dyeing, mercerising, tanning, brewing, alcohol distillation, and all experimental processes. Copies of the draft regulations may be obtained from the Factory Department, Home Office, S.W. 1, and objections must be lodged within 30 days of December 24.

**ORDERS CANCELLED.**—The Oil and Fat Compound (Licensing of Manufacturers and Requisition) Order, 1918, the Seeds, Oils and Fats Order, 1919, and the Cattle Cakes and Meals (Licensing) Order, 1919, have been revoked, as from December 23.

**INCREASED WAGES IN THE CHEMICAL INDUSTRY.**—The Industrial Court has awarded to plain time-workers increases in wages which amount to, in general, 39s. 6d. a week plus 12½ per cent. on total earnings over the pre-war rates, and equivalent advances to pieceworkers.

## OFFICIAL TRADE INTELLIGENCE.

(From the *Board of Trade Journal* for December 23 and 30, and January 6.)

### OPENINGS FOR BRITISH TRADE.

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W. 1, from firms, agents or individuals who desire to represent U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number:—

Locality of Firm or Agent.	Materials.	Reference Number.
Australia .. ..	Chemicals, oils, grease .. ..	891
" .. ..	Crockery, glassware .. ..	3
Canada .. ..	Copper and brass rods, tubes and sheets, tinplate .. ..	858
" .. ..	Tiles, fire-brick, glass, marble, slate .. ..	4
" .. ..	Soda ash .. ..	865
Hong Kong .. ..	Chemicals .. ..	865
New Zealand .. ..	Chemicals, dyes, paper .. ..	7
South Africa .. ..	Solid-drawn hard copper tubing .. ..	860
" .. ..	Antifriction grease, paint, glass, wrapping paper .. ..	861
Belgium .. ..	Glue, glass-paper, varnish .. ..	869
" .. ..	Bichromate and permanganate of potash, sodium bicarbonate, paraffin wax, petroleum jelly .. ..	870
" .. ..	Chemical and pharmaceutical products, materials for the manufacture of chocolate and perfumery .. ..	11
Bulgaria .. ..	Articles in demand include oils, grease, emery, iron, cement, mineral oils, copper sulphate, paper, window glass, dyes, colours, chemicals, lead, copper, tinplate, and wire .. ..	873
France .. ..	Chemicals, cattle foods, fertilisers .. ..	873
Greece .. ..	Glass and china ware, leather, dyes, and photographic goods .. ..	13a
Italy .. ..	Chemical and pharmaceutical products .. ..	879
" .. ..	Tinplate, steel and iron tubes .. ..	14
Latvia .. ..	Copper tubing .. ..	15
Switzerland & Italy .. ..	Heavy and pharmaceutical chemicals, oils, fats .. ..	883
Morocco .. ..	China and glass ware .. ..	22a
China .. ..	Scientific supplies .. ..	898
Japan .. ..	Magnesium, aluminium, lead, tin, tinplate, steel sheets, caustic soda, soda ash, carbolic acid, fertilisers .. ..	899
Brazil .. ..	Tinplate, tin, metals, chemical and pharmaceutical products, cement, paper .. ..	93
Chile .. ..	Glassware, pottery, explosives, paper .. ..	30
Mexico .. ..	Drugs, perfumes, leather (catalogues of) .. ..	887
Peru, Bolivia, Ecuador .. ..	Chocolate .. ..	888
United States .. ..	Antimony .. ..	885
" .. ..	Metals .. ..	25
" .. ..	Glassware .. ..	27

\* The Canadian Government Trade Commissioner, 73, Basinghall Street, London, E.C. 2.

† The High Commissioner for Canada, 19, Victoria Street, London, S.W. 1.

**MARKET SOUGHT.**—A firm in Japan wishes to get into touch with U.K. importers of camphor, menthol, vegetable and fish oils, starch, ground nuts, and isinglass. [899]

### TARIFF. CUSTOMS. EXCISE.

**Australia.**—The embargo on the export of goods to Germany, Austria, Hungary, Turkey and Bulgaria has been removed.

**Austria.**—Customs duties when paid in bank notes have to be paid at 40 times the rates prescribed by the Tariff as from December 1.

**Belgium.**—Export licences are no longer required for steel ingots, etc., fire-bricks, chemical manures

(with certain exceptions), certain oil-seeds, bone and bone powder, newsprint paper, and natural phosphate.

*British Guiana.*—The prohibition of the import of dyes and dyestuffs not manufactured within the British Empire has been temporarily withdrawn.

*British Honduras.*—The new customs tariff came into force on October 12, 1920.

Among the articles exempted from duty are coconuts, cohune nuts, certain drugs, charcoal, raw rubber, balata, chicle, raw hides and skins, horns, bones, fertilisers, fungicides, insecticides, copra, quinine, sand, slate, scientific apparatus, certain tanning and dyeing materials, tonca beans, serums, and radium.

The articles admitted duty free under the British Preferential Tariff include explosives (except sporting), tiles, bricks, cement, barbed wire, unskimmed condensed milk, fuel and lubricating oils, newsprint paper, and rock salt.

*Canada.*—Drawback of 99 per cent. of the duties paid is now allowed on the materials used in the manufacture of margarine.

The customs duty on condensed milk has been increased; and the "luxury" taxes, with certain exceptions, have been repealed.

*Colombia.*—The export of gold is now permitted.

*Costa Rica.*—The text of the revised consular regulations of Costa Rica may be seen at the Department, 18, Queen Anne's Gate, S.W. 1.

*Denmark.*—Export prohibitions have been removed from, *inter alia*, cement, gypsum, prepared hides and skins, leather, old cast iron, fertilisers (except potash compounds), starch, soda, and tar.

*Egypt.*—The issue for January 6 contains a list of the revised tariff valuations for use in assessing the duties on imported non-ferrous metals.

*Germany.*—The detailed list of "controlled" imports may be seen at the Department, 18, Queen Anne's Gate, S.W. 1.

The import of waste paper, paper clippings, and paper, pasteboard and pasteboard wares suitable only for re-pulping is allowed without licence.

Among the articles for which no import licence is required are raw cocoa, rubber, gutta-percha, balata, certain feeding stuffs, asbestos, and some glass wares.

No export licence is required for ambergris, night tapers of waxed thread, natural spring salts, raw silk, wax cloth, emery cloth, carbons for arc lamps, certain wares of talc, asphalt and glass, spun silver and aluminium, and threads of common metals.

*Italy.*—The surcharge on customs duties when paid in paper money is fixed at 200 per cent. as from December 15, 1920.

Export licences are no longer required for, *inter alia*, bichromates of soda and potash, dinitrotoluol, milk sugar, certain medicines, tin, aluminium and zinc ash. Export licences are now required for linseed and maize cakes, pyrites, etc.

*Lithuania.*—The export duty on linseed has been increased from 25 to 30 marks per pound.

*Luxemburg.*—The export is now permitted of tin, pottery, earthenware, ceramic wares, india-rubber and india-rubber wares. An import licence is still required for cement, and the import of saccharin is prohibited.

*Mexico.*—Recent modifications of the customs tariff are set out in the issue for January 6.

The export duty on copper has been removed.

*New Zealand (Samoa).*—The customs tariff is set out in the issue for January 6.

*Northern Rhodesia.*—The customs and excise duties on spirits have been amended as from October 16, 1920.

The importation of certain dyes and dyestuffs not manufactured within the British Empire is prohibited.

*St. Vincent.*—The prohibition of the export of hides and skins has been revoked, and that on the

import of certain dyes has been temporarily suspended.

*Serb-Croat-Slovene State.*—The period during which goods may remain in customs warehouses has been reduced to five days, and outside the warehouse to ten days, after which the goods will be sold.

*Sweden.*—Among the articles still subject to export licence are crude phosphate (except apatite), asbestos, asphalt, coloura, lard, tallow, margarine, starch, cattle foods, milk powder, yeast, sugar, syrup, glucose, colouring matter, certain hides and leathers, vegetable fibres, certain metals and alloys, mineral oils, vegetable oils (with some exceptions), varnish, putty, caustic potash, potash salts, tin salts, indigo, Chilean nitrate, and animal waste.

*Switzerland.*—Special export licences are still required for, *inter alia*, cocoa, chocolate, sugar, preserved milk, oil cake, hides, skins, sole leather, charcoal, fibrous materials for the manufacture of paper, rubber, gutta-percha, porcelain insulators, certain kinds of glass, certain ores, metals and metal scraps, alkaloids, saccharin, certain chemicals, pitch, sulphur, turpentine, tar, caustic potash and soda, dyes, colours, colour varnish, and mineral oils.

*Tripolitania and Cyrenaica.*—The import duty on spirits has been increased.

*Tunis.*—A special tax of 2 fr. per metric ton is payable on exports of phosphates, in addition to the duty of 50 cts. per metric ton.

## TRADE NOTES.

### BRITISH.

*Cyprus in 1919-20.*—Cyprus is essentially an agricultural country, the chief products being fruit, cotton, sumac, and castor oil. Good results have been obtained in the cultivation of beetroot, but experimental plots of sugar-cane were a failure. Wine-making is, however, very prosperous. The forests constitute very valuable resources, and experiments on the distillation of forest produce for the production of pyroligneous acid have been continued. Difficulties in obtaining supplies have hindered mining operations, but these have been overcome and preparations for working on a large scale have begun. The Cyprus Mines Corporation has extracted 820 tons of cupriferous iron pyrites. A local syndicate, which acquired the property of the Cyprian Mining Co., was actively engaged in mining asbestos at Amianos. Gypsum and amber were also produced.

The imports were valued at £1,525,427 (£1,013,532 in 1918), and were chiefly of British origin (81·2 per cent.). Exports amounting to £1,371,580 (£10,235 increase) were directed mainly to British possessions (£664,424) and the United Kingdom (£313,455). The export of raw amber increased from 53 tons to 2785 tons, and the trade is rapidly increasing; the United States took 1254 t. of amber, Italy 986 t., and the United Kingdom 335 t.—(*Col. Rep.-Ann.*, No. 1046, Nov., 1920.)

### FOREIGN.

*Soda Prices in Germany.*—The Soda Syndicate has reduced the prices of soda products by 5 to 10 per cent.—(*Chem. Ind.*, Dec. 8, 1920.)

*Japanese Trade in Caustic Potash.*—The annual pre-war requirement of Japan in caustic potash was about 11,180 long tons, half of which was supplied by home producers, and the rest mainly by England and the United States. The cessation of all imports during the war led to a great development of the home industry, and at the present time the annual production is about 13,400 t., though 22,000 t. might be produced by effecting improve-

ments in manufacture. In consequence of speculative buying the home market is flooded with supplies, and manufacturers are asking for a protective duty; but it is unlikely that this would lead to improvement so long as the raw material used remains so poor.—(*Chem. Ind.*, Dec. 8, 1920.)

**Peruvian Exports of Cochineal.**—Ayacucho and Huanuco are the chief cochineal-producing districts of Peru. The production, which before the war was bought by German houses, is now practically all being sold to the United States. The total export in 1917 was 22,193 kg., of which the United States took 5385 kg., and Great Britain 6672 kg.; in 1918 the former country took 33,165 kg. Cochineal is exported to the United States and to Europe through the port of Callao.—(*Rev. Prod. Chim.*, Nov. 15, 1920.)

## REPORT.

**REPORT OF THE LUBRICANTS AND LUBRICATION INQUIRY COMMITTEE.** *Department of Scientific and Industrial Research Advisory Council.* Pp. 126. London: H.M. Stationery Office. 1920. Price 2s. 6d.

Before the war the annual expenditure on lubricants in this country was six million pounds, and it was estimated that a saving of one to two million pounds could be effected if a systematic investigation on the subject was instituted and the results were made freely accessible to the public. The Advisory Council thereupon decided to set up a Lubricants Committee with the following reference:—"To prepare a memorandum on the field for research containing an analysis of the problems involved, together with a suggested scheme of research which would be likely to lead to valuable results." The committee was presided over by Mr. S. B. Donkin, and contained such well-known authorities as Archbutt, Boys, Deeley, Donnan, Sir C. Parsons, and Lord Rayleigh. A special sub-committee has compiled an exhaustive bibliography of the chemical, physical, and engineering aspects of lubrication which will be published shortly. A considerable amount of research is detailed in the report under notice, notably on the quality of "oiliness," which has been attributed to lubricating oils by so many workers. The National Physical Laboratory has, in particular, been active in the prosecution of these inquiries, which, however, have not yet been brought to a conclusion. Certain material and special aspects of lubrication were brought to the notice of the committee and have necessitated individual investigations and reports which in two cases (*Cutting Lubricants and Solid Lubricants*) have been issued to the public.

Of the research work carried out at Teddington may be mentioned (a) Tests with the Lanchester worm gear, including a variety of mineral, animal, and vegetable oils with and without the addition of graphite and "oildag"; (b) the effect of pressure on viscosity; (c) determination of the physical constants of oily and non-oily liquids; (d) special methods for the determination of viscosity of oils under pressure in bearings (Boys and Parsons); (e) Deeley's oil-testing apparatus; and (f) the cup and ball viscosimeter. A series of recommendations for future research is tabulated from chemical, physical, and engineering points of view.

Among the appendices may be noted Faust's paper on viscosity at high pressure, a series of researches by Hyde on the Lanchester gear with a variety of lubricants, papers by Parsons and Boys, Deeley and Thomson on viscosity determination, and notes on "oildag."

## REVIEWS.

### THE ART OF TECHNICAL WRITING.

**TECHNICAL WRITING.** By T. A. RICKARD. Pp. iv.+178. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1920.) Price 8s. net.

The number of books on English composition is legion; among the latest and best is the "Art of Writing," by Sir Arthur Quiller-Couch. But there is still room for a book on the art of writing the literature of science, and particularly the art of putting together communications intended for the pages of scientific and technical journals. It may be alleged that this is literature of no very high order. Elia would possibly have classed books on technology as among books that were no books. But Lamb lived in an unscientific age. Other times, other views. We live in an age of science, and science needs the proper expression of its message as much as any other branch of intellectual activity. "Science and literature," said Huxley, "are not two things, but two sides of the same thing." But, of course, the literature we class as belles-lettres—the literature of rhetoric, poetry, fiction, philology—is characterised by other attributes than those which distinguish the literature of science. There should be no occasion for passion or the play of emotion in a purely scientific disquisition. Thoughts that breathe are to be welcomed, but words that burn are not, and purple patches are out of place. But the fundamental principles of the art of literary expression are common to every branch of literature, and that of science, whether pure or applied, needs them no less than any other.

Men of science, as a class, are not infrequently reproached by humanists for their apparent disregard in their written work of all literary form. "The man of science," said Sir James Barrie, "appears to be the only man who has something to say, just now—and the only man who does not know how to say it." The generalisation is, of course, too sweeping, but every editor of a scientific journal will be ready to admit that there is substantial ground for its partial truth. The author of the book before us, at least, has no doubt of the fact as regards the technologist, and especially the engineer, who, he finds, "does bungle language deplorably. He makes a fetish of efficiency, yet he shows no regard for the effective use of one of his most important tools—the pen; he believes devoutly in accuracy, yet he employs a weapon of precision as carelessly as a small boy handles a gun." But it is not only the engineer who is thus at fault. Technical students as a class are equally to blame. This, to judge from the authorities the author cites, is widely recognised in America. Its cause, in the opinion of the Senate of the University of California, which appears to have gravely considered the matter, is mainly due "to the unedifying influence of myriad examples of poor English" in the local press. The newspapers the student reads tend to spoil his taste for good English. In addition, he is affected by "a certain public prejudice against correctness of expression," presumably fostered by the local press, which in later life, when he is in "daily contact with illiterate men, inclines him to careless speech and slovenly writing." "The uneducated are inclined to resent any speech more polished than their own."

It is not for us to criticise a finding thus deliberately reached. American journalism, no doubt, has much to answer for. As a rule, its literary quality is not high, although all editors would not concur with the Denver journalist quoted by our author, who maintained that literary form is "a mere frill," and that the main business is "to get there"—no matter how clumsily!



Examples of solecisms in the American press are quoted that would disgrace a schoolboy. "Our grandfathers used to read the Bible daily; we read the daily newspaper." "The time given to the piffle of the press is lamentable."

But why behold we the mote that is in our brother's eye, but consider not the beam that is in our own eye? This book, although based upon lectures delivered to American students, and primarily addressed to an American public, contains truths which we on this side of the Atlantic may well lay to heart. Journalism is not confined to the United States. We, too, have newspapers which as regards their literary quality not only follow but even seek to better the example of the Denver editor. It may be that their influence on the character of technical writing is inconsiderable, but the daily reading of "piffle" blunts our appreciation of good English and insensibly lowers our standard of excellence. Style can only be absorbed subconsciously by continued contact with the best authors.

Mr. Rickard's criticism, like all useful criticism, is constructive. If he points out a fault he shows how it should be amended. He enunciates rules and illustrates their application. It is remarkable how much he manages to pack into his 178 small octavo pages. He recognises that instruction by precept is long; by example, short and effectual. Considerations of space preclude any detailed attempt to deal even with the general principles he inculcates, but it may be of use to give a short summary of his teaching. Much of it, at first sight, seems almost axiomatic and so obvious that it hardly needs to be stated. But so are the Ten Commandments, yet it is found desirable to repeat them at least once a week. Trite as are the rules of good writing, many of them are more constantly honoured in the breach than in the observance.

Mr. Rickard's golden rule is: *Remember the reader*. Our first aim should be to be understood. Technical writing is the precise expression of special knowledge. We are enjoined to be natural; it is the key-note of good writing, as of good manners. The skilful use of the pen, *pace* Dogberry, is not a gift of nature. To write naturally you must exercise the faculty of writing until it becomes flexible and strong. Good writing depends not so much upon a large vocabulary as upon the choice of words. The dominant fault of the incapable writer is the employment of too many words. Verbosity is a sign of carelessness or of lack of time for proper time. A conscientious author has been known to apologise for the length of his book, like Pliny for the length of his letter, because he had not time to make it shorter. Don't be tempted into the use of high-sounding terms that may serve to cover ignorance. "I have often observed," said Macaulay, "that a fine Greek compound is an excellent substitute for a reason." Greek terms, like razors, are not to be flourished recklessly. Do not use words of classical origin unless you know their meaning and have some knowledge of their etymology. Clearness of statement necessitates precision. The technical term is a word of precision, but let precision be proportioned to accuracy. Do not try to be precise in matters that do not admit of accuracy. The technical writer must be sincere; sincerity is the first essential of all good work. Suit the style to the purpose; fitness is the essence of good taste. Discard the trivial words that are constantly at your elbow; brush aside a host of rapid superlatives. "The superlative," said Metternich, "is the mark of fools." For example, the little word "very" can be deleted nine times out of ten: it is an impediment to terse and perspicuous writing. "Very" makes a strong adjective but a weak adverb; if you employ "very"

infrequently, you will find it very useful on occasion. The secret of a vigorous style is the rejection of the superfluous word, but do not hesitate to repeat a word in order to make yourself clear. Perspicuity is the better part of elegance. A doubtful meaning is worse than a grammatical error. Good writing calls for care—persistent care; it calls for a literary conscience that refuses to be satisfied with unfinished work. As the Latin tag has it: *Sape stylum veritas*.

These aphorisms, culled from various sections of this book, and largely in the author's own words, will serve to show the character of his work. It deserves to be read, marked, and learned by all who write for the technical press. We may not always agree with his judgments. There are no final or absolute standards in the art of writing, and even the greatest masters of prose may sometimes, like Homer, be thought to nod. But Mr. Rickard has at least the courage of his convictions, and his sincerity and candour merit our appreciation.

T. E. THORPE.

HANDBOOK OF PATENT LAW OF ALL COUNTRIES. By W. A. THOMPSON. Eighteenth edition. Pp. vi.+157. (London: Stevens and Sons, Ltd. 1920.) Price 6s. net.

This is a new edition, considerably revised in view of the extensive modifications which the patent law of the world has undergone during the years of war, of a well-known book. It is intended for the assistance of the public, and of inventors in particular, and is compiled from that point of view. The summary given of patent law is terse and popular in form. In substance it covers the points most likely to present themselves to an intending patentee. It is written in a breezy and readable style. Although the title refers only to patent law, the subject matter combines the cognate subjects of Trade Marks, Designs and Copyright registration, these subordinate subjects, however, being dealt with less systematically than the main theme.

In writing on such a subject as English patent law, it is almost impossible to deal satisfactorily with the historical aspects of the subjects, themselves extremely well worth study, in a few words. Thus a sentence such as the following:—"As a rule it is undesirable to make a body corporate one of the applicants, as it is not quite certain whether in such case every member of the said body has not an independent right to use the invention"—is probably misleading. The practice of making limited companies parties to patent applications is a very common one, and the suggestion that every member of such a company has an independent right to use the invention would certainly be received with considerable astonishment by any modern court. There are, no doubt, old dicta, applied primarily to franchises differing in kind from those conferred by a patent grant, which might be quoted in this connexion, but there is no real doubt that they would be promptly swept aside. So again the remark that:—"In old times it was held unconstitutional for the King to grant a monopoly to a foreigner resident abroad"—is a proposition which stands much in need of verification if it is to convey any just impression of the course of development of English law. Here condensation is hardly compatible with the usefully clear statement of the point intended to be made. On the other hand, a very slight amendment would set right the sentence which runs thus:—"The word" (invention) "is derived primarily, as is well known, from the latin *in venire* to come in."

These, however, are small matters; they do not affect the value of Mr. Thompson's book, which exhibits in a very compendious form the outlines of patent law in Great Britain, the British colonies



and dependencies and all foreign countries that grant patents. Of special interest to its readers will be the accessory information, such as the dates of the various patent laws digested and the populations concerned, which are systematically given. We miss from this what was very useful information in the earlier editions of the book, that is to say, notes of the patent office fees in the various countries.

There is one point of very general interest in respect of which Mr. Thompson has made himself the mouthpiece of a curiously mistaken notion about the British Patent Office. On page 19, speaking of the renewal fees payable under British patents, he says:—"The Board of Trade has power to reduce these taxes, which are much too high and bring large profit to the Treasury." The fact is that the Patent Office, although at present safely paying its way, can hardly be regarded as a revenue producing department at all. The Comptroller General's report for the year 1919 shows a total income from all sources, including profit on the sales of specifications, etc., of £404,474, whilst the expenses of running the establishment were £350,150, showing a profit income of £54,324, which is no more than would be expected to be realised on a commercial venture of corresponding magnitude. No doubt there are risks attending ordinary commercial enterprises from which the business of conducting the Patent Office is free, and the 12 per cent. profit balance may be looked upon as a tax charged upon patentees; but it certainly is a very moderate tax and, having regard to the fact that no patentee pays it unless it is worth his while to do so, the complaint in these days of inevitably high taxation is a little unworthy.

J. W. GORDON.

THE VOLATILE OILS. By E. GILDEMEISTER and FR. HOFFMANN. Second Edition by E. GILDEMEISTER. Authorised translation by EDWARD KREMER. Second Volume. Pp. xx.+686. (London: Longmans, Green and Co., 1920.) Price 32s. net.

Gildemeister and Hoffmann's work on "Essential Oils" is so well known as to need no recommendation from reviewers. The present volume, however, comes into existence under very unfortunate circumstances. Written mainly in 1912 in German, it was published in Germany in 1913. It appears to have been translated between 1913 and 1916 by Dr. Kremers, printed in 1916, and published in 1920. The translator has allowed himself to translate Dr. Gildemeister's preface, dated "Miltitz, near Leipzig, June, 1913," as "Miltitz, near Leipzig, June, 1916," which we think should not have been done, as no attempt has been made either by author or translator to bring the work beyond the original date of 1913.

The present volume follows the plan of Volume I., the essential oils being enumerated in the order of Engler's "Syllabus der Pflanzen-familien," and the oils dealt with are those up to and including those of the *Zygophyllaceæ* and some of those of the *Rutaceæ*.

If this work were being reviewed in 1913, the reviewer's task would be an easy one, and nothing but praise and commendation could be accorded to it. Unfortunately it is 1920, and the book is so considerably out of date that we think it a pity that it should not have at least been brought up to the latest date possible by the translator. The reason for this serious shortcoming is obvious, but none the less, although every chemist interested in essential oils will welcome the book, the work done between 1913 and 1920 in this field is so great that the book loses very much of its utility.

To illustrate this, attention may be called to a few only of the more important omissions as types of those occurring throughout the volume.

Under the various cedar-wood oils no account is to be found of the work of Roberts on the oil of *Cedrus deodara* (1916), and the interesting researches of Schimmel and Co. on the ketone contained therein (1917). The latest work on cryptomeria oil dealt with is that of Kimura (*cf. Berichte*, 1909, p. 372); but in 1916 So Uchida discovered a crystalline diterpene in the oil, and published interesting details of the odorous alcohol present in the oil. The latest work on the *chamæcyparis* oils is dated 1910, so that Schorger's work (1914) and Uchida's (1916) are not mentioned. Still, keeping to the *Pinaceæ*, we find no references to Parry's work on "Russian Oil of Turpentine," which was published in 1912, nor, of course, to the exhaustive reports on the various turpentine and wood-turpentine oils of the different pines in the United States made by Schorger and Adams in 1915.

Turning to the oils of the *Gramineæ*, we are pleased to notice that at last we have a definite agreement that "the quality of citronella oil is best ascertained by the quantitative determination of the acetylisable constituents." Those who have perused Messrs. Schimmel's reports for the past few years will remember how strenuously certain British analysts insisted upon this for years, and how vigorously Messrs. Schimmel and Co. opposed it. It is, however, regrettable that the acetylation is directed to be carried out with equal volumes of the oil and acetic anhydride. This will always give low results, especially with oils of the Java type and 10 c.c. of the oil should be treated with 15 c.c. of acetic anhydride. The data concerning ginger oil are, from the cause indicated above, quite out of date, as Brooks in 1916 identified in it the following substances:—methylheptenone, nonyl aldehyde, linalol, and zingiberol.

A few words only are devoted to the important oils of False Cuhebs. This subject was exhaustively dealt with by Oranje in 1914, and any account must necessarily be quite incomplete without reference to this work. Equally true is it that Rabaki's interesting work on "Oil of Hops" (1914) is unnoted; the oil of *Cinnamomum Oliveri* is dealt with on the basis of Baker's work of 1897, which has been superseded and partially contradicted by that of Hargreaves in 1916; oil of massoy bark is stated to contain safrol, Messrs. Schimmel and Co. being the authority; whereas in 1917 the same authority stated that safrol could not be detected. The above are merely a few examples of the inevitable result of publishing in 1920 a work written in 1913 on a rapidly moving branch of chemistry. Due allowance having been made for this circumstance and judging the book as in 1913, one may accurately describe it as being particularly free from errors of any kind.

E. J. PARRY.

## PUBLICATIONS RECEIVED.

ORGANIC MEDICINAL CHEMICALS. By M. BARROW-CLIFF and F. H. CARR. *Industrial Chemistry Series*, edited by S. RIDEAL. Pp. 381. (London: Ballière, Tindall and Cox. 1920.) Price 15s.

INDIA-RUBBER GOODS MANUFACTURE. By "FACTORY MANAGER." Pp. 496. (London: MacLaren and Sons, Ltd. 1920.) Price 32s. 6d.

A TEXT BOOK OF CHEMICAL ENGINEERING. By E. HART. Pp. 211. (Easton, Pa.: The Chemical Publishing Co.; London: Williams and Norgate. 1920.) Price \$4.

THE ANALYST'S LABORATORY COMPANION. By A. E. JOHNSON. Fifth edition. Pp. 176. (London: J. and A. Churchill. 1920.) Price 10s. 6d.

## THE CONDITION AND PROSPECTS OF THE BRITISH CERAMIC INDUSTRY.

J. A. AUDLEY.

Less than a year ago an Australian buyer entered the office of a well-known North Staffordshire earthenware manufactory, laid down a cheque for £10,000, and said in effect, "There you are! What can you do for us?" It can readily be understood that the manufacturers' representative keenly regretted the necessity of having to explain that it was impossible to do anything at all for at least twelve months. Within the last two or three years incidents of the same kind, though varying as regards the money value concerned, have been by no means infrequent in the ceramic industry. This state of affairs could not, in the nature of things, last indefinitely, and as regards some classes of ceramic products new conditions have already arisen.

It will be remembered that one outstanding consequence of the great war was an enormous dislocation of international trade and commerce. Many things formerly obtained mostly from abroad, and which were included in the category of necessities, had to be provided somehow in our own country. The manufacture of articles by people with limited experience—workers and supervisors alike—and often under conditions of urgency requiring the promptest possible delivery of the goods, did not tend to the production of the highest quality. The same was largely true also of ordinary commodities, because the ready sale at remunerative prices for home consumption of practically all classes of goods left no incentive to the manufacturers to seek improvements in design, shape, decoration, or general quality, unless such happened to be intimately associated with increased output. Ceramic products were no exception to the general rule, and buyers have been to an unusual degree at the mercy of the manufacturers. The long period of delay in effecting world settlement has greatly helped to prevent trade from resuming or even approaching its normal course, and the well-meant attempts to establish in this country a state of preparedness or possible future wars—having regard to all the existing circumstances it might not inaptly be expressed as for probable future wars—has militated against any substantial reduction in prices. These considerations, though general in their application, carry greater force in some directions than in others.

The china doll trade affords an instructive example. When few or no dolls were obtainable from abroad, home products supplied the market, but it is well understood that, with rare exceptions, they were decidedly inferior to similar articles formerly imported, besides being more expensive. Instead of seizing the opportunity to establish good models and to produce superior ware from them, the manufacturers were content to pile up profits on easily-made but inartistic goods. The inevitable result is that when dolls from abroad are available, they are found to be not only cheaper, but also much better than those made in this country. So too with fancy ware, which sold well while there were no competitors and money was fairly plentiful, but failed lamentably when faced with serious competition. This likewise was due to the fact that no effort was made to adapt the ware to modern requirements as regards artistic and other qualities, and the sale accordingly fell away. There is still a demand for fancy goods, but not for inferior yet expensive ware

which may be ranged in that class. As the world shortage of supplies gradually becomes satisfied, and as international trade relations with Russia, Germany, and other countries get restored, the vegetable and mineral exports from these places will have a powerful effect on the prices of similar commodities in America, and this in turn will affect the prices of pottery, etc., purchased in America from this country and elsewhere. It would therefore be wise for manufacturers to look forward and prepare for the coming fall in prices, to concentrate their efforts on lines that are likely to be in continued demand, avoiding particularly articles which will not be able to compete successfully with cheaper similar goods from other countries. This is not an absolute necessity in the economic situation at the moment, but the rapid changes and tendencies of that situation should be carefully studied, and suitable action taken from time to time in order to be prepared for developments. In this way the probable ill effects of the threatened fall in prices—or, at any rate, the worst of them—would be averted.

The supply of fuel has under present conditions been very far from satisfactory, especially as regards quality, and the restlessness among the coal miners has introduced an additional adverse factor, but the latter difficulty is now causing less anxiety, and improvements in other directions are also hoped for shortly.

The modern tendency to co-operation, which has already proved beneficial to the manufacturers in many industries, has within the last two or three years made such progress among pottery manufacturers that a British Pottery Manufacturers' Federation has been formed, and has already amply justified its existence, though serious difficulties still lie ahead. It includes most of the manufacturing firms, but not all. Before the war, wages in the pottery industry were notoriously low. The very abnormal conditions which resulted during and after the war provided a good opportunity for a great improvement to be made in this respect without causing any serious inconvenience. The poor pre-war wages scale was by some attributed to foreign competition, more especially on the part of Germany and Austria. This view was apparently negated by the fact that by means of mass production of a few specialties, using modern appliances and methods, several British firms had actually competed successfully against German firms in Germany itself, in spite of tariffs and other obstacles. A much more probable explanation is that given by the representative of a prominent manufacturing firm, who candidly expressed his belief that it resulted from bad organisation.

There is less difficulty about a general rise in prices than in a fall. The inevitable fall in prices which will sooner or later follow a continuous decline in trade—the invariable sequence after the rise in prices which accompanies great wars, as far back as the wars of Napoleon at least, though the prices have never gone down to the pre-war level—will provide a troublesome problem for the industry, because the smaller manufacturers are much weaker economically than the larger firms, and any weakening on the part of the smaller firms will to some extent weaken the stronger position of the larger manufacturers. This difficulty might be met by the adoption on the part of the Manufacturers' Federation of appropriate schemes of selling: co-operative selling would be advantageous, especially for the smaller firms. This, however, can hardly be accomplished effectually unless standardised goods are produced under a system of scientific costing. Present prices were arrived at by merely adding percentages to pre-war prices,

but the latter differed from factory to factory according to the more or less economical production of the several articles, or to the varying amounts of the profits reserved by the different manufacturers.

British ceramic products, so far at least as the better class articles are concerned, are distinguished by the good potting of the body composition, and by the excellence of the glaze and colouring. So long as these features are maintained, the ware will always be in demand abroad, but the extent of that demand will depend largely on other considerations, notably on the selling price.

Chemical porcelain was entirely imported into Great Britain before the war; it was one of the few ceramic lines not produced in this country. When the supplies were no longer available, it became necessary to provide a substitute. A number of firms carried out experimental work, and most of them obtained encouraging results; of these firms the Royal Worcester Porcelain Co., Ltd., and Messrs. Doulton and Co., Ltd., persevered so far as to supply in quantity various articles for laboratory use, and continue to do so. Some of these British products were favourably reported on by the National Physical Laboratory. With reasonable care they may be used for most of the purposes of laboratory porcelain ware, but they can scarcely be regarded as more than serviceable substitutes, and it would not be correct to state that they are equal to the Berlin porcelain, for example, which stands very severe heating tests. The British ware has not the same composition, nor are the firing conditions the same as for the continental hard porcelain, and the continuance of a demand for it must presumably depend on the maintenance of a high quality, coupled with the ability to compete successfully as regards price with the foreign hard porcelain.

At the research establishment connected with the Stoke-on-Trent Pottery School, investigations have been carried on for about three years under the direction of Dr. J. W. Mellor and Mr. Bernard Moore, with a view to the production of hard porcelain. A type of hard porcelain suitable for domestic ware, and made from British materials by essentially British methods, constitutes one gratifying result of this work. Pieces have been produced exhibiting a wide range of colour decoration, and some manufacturers have undertaken to produce this ware on a commercial scale, but the prevalent abnormal trade conditions have for the time being hindered developments. True hard porcelain has also been made on a small scale at the same institution, but in this direction also no further development has yet taken place.

Brief mention may here be made of the "Vitreon" ware placed on the market by Messrs. Shanks and Co., Ltd. This is a fine white stoneware of great strength and impermeability, which is largely used for various articles needed in chemical manufacturing processes, and seems likely to compete successfully with the best German stoneware.

**ROYAL SANITARY INSTITUTE.**—The Henry Saxton Snell Prize of fifty guineas and the medal of the Institute are offered for an essay on "Suggestions for a System of Central Hot-Water Supply and Heating, adapted to Modern Housing Schemes, and to existing Groups of Houses." The essays, which should deal with the central installation, the distribution and conservation of the heat, the provision of continued supply during repair, the cost and combination with other services, must be sent before August 31, 1921, to the Secretary of the Institute, 90, Buckingham Palace Road, London, S.W., from whom all particulars can be obtained.

## CHEMISTRY OF CLARIFICATION IN PLANTATION WHITE SUGAR MANUFACTURE.

J. P. OGILVIE.

In the Board of Trade returns relating to the importation of goods into the United Kingdom, all white sugars are collected for the sake of convenience under the heading of "refined." It is to be noted, however, that a good proportion of the white sugars bought by us since 1914 has been of the grade known as "plantation white," that is, a product made in the factory directly from the cane juice without being submitted to the refining process (which comprises re-solution, decolorising by passing through animal charcoal, and recrystallising). Such a tropical white sugar, though generally lacking the beautiful appearance of the best refined, has a ready sale, not only for use in the home, but largely in the confectionery, jam, biscuit, and condensed milk industries.

Undoubtedly, the clarification of the juice is the most important operation in making plantation white sugar. It is, therefore, of interest to notice briefly the essential differences between the procedure followed and that used in manufacturing the lower grade product requiring to be refined before it can be consumed.<sup>1</sup>

Cane juice as it comes from the mills is a light grey to dark-green turbid and frothy fluid, which, in addition to sucrose and reducing sugars, contains in solution free organic acids (as acetic, glycolic, oxalic, malic, etc.), organic and inorganic salts, gums and pectins, proteins, such colouring matters as anthocyanin, saccharatin, and the ferric-polyphenol compounds recently investigated by Zerban.<sup>2</sup> It also holds in suspension very fine particles of the crushed cane (so-called "cush-cush," or *bagacillo*), chlorophyll, wax, and lastly a little sand and clay, all of which solid impurities have escaped retention by the strainers of very fine mesh through which the liquid coming from the mills is preliminarily passed. Owing to its content in gums and pectins, it is unfortunately impracticable to filter the raw juice, either through cloth, sand, bagasse, or other medium.

In making ordinary raw sugar, the process of clarification adopted consists briefly in adding milk of lime to slight alkalinity or to neutrality (to litmus), heating to about 90° C., and allowing to subside in suitable settling tanks, the juice decanted from the scums and "mud" being sent to the multiple-effect evaporators for concentration to a syrup, from which the sugar is subsequently crystallised in the vacuum pan. This treatment is far from efficient in eliminating the soluble and insoluble impurities. It results in the free acids being neutralised, the proteins and nucleins being coagulated, and most of the floating "cush-cush" wax and clay being enveloped in the precipitate. Pectins and gums, it should be noted, are only partly eliminated by the small quantity of lime used. Regarding the colouring matters, the chlorophyll present in suspension is removed with the other floating substances, but the anthocyanin pigments coming from the bark of the cane remain to a large extent in the juice, adding very materially to its colour, especially if the variety of the cane being crushed is a very dark one. Saccharatin, which is a soluble pigment, and possesses the character of an indicator, being intensely yellow in alkaline solution, and colourless in an

<sup>1</sup> A very good account of tropical white sugar manufacture is given by Harloff and Schmidt in their "Handbuch der Tropen Zuckerfabrikation," which has just reached its 4th edition. An English translation of this work (from the 2nd edition) appeared in 1913.

<sup>2</sup> This J., 1918, 778a; 1919, 192a.

acid medium, likewise remains; as do also the ferric-polyphenol compounds, which similarly contribute largely to the colour of the juice.

This method of treating with a small amount of lime, generally known as the "defecation process," by no means exerts the maximum clarifying effect. It gives a juice containing a certain amount of solid impurities in suspension, which accumulate in a rather dark syrup, and later during graining in the vacuum pan become incorporated between the lamellæ of the crystals, imparting to them a dirty colour incapable of removal by washing in the centrifugal machines with syrup, water, or steam. Not the least of the several defects of the defecation process, however, is that it may actually permit the formation of colouring matter. This is largely due (though apparently not entirely)<sup>1</sup> to the rather ready decomposition of dextrose and levulose (comprising the so-called "glucose") at temperatures above 55° C., especially in an alkaline medium, viscous, dark-brown calcium salts of glucinic and saccharinic acids being produced. These decomposition products of "glucose" are probably the most inimical substances with which one has to deal in white sugar manufacture. They are only partly and temporarily bleached by sulphur dioxide, or by sodium hydrosulphite, and being very soluble persist through the several stages of manufacture, finally imparting a faint tinge of colour to the finished white sugar. Their formation must consequently be avoided as far as possible.

In white sugar manufacture, on the other hand, the method of clarification chosen is much more thorough, especially in regard to the elimination of the pectins and gums. Processes have been adopted which produce such a clarifying effect that these troublesome constituents are eliminated to a sufficient extent to enable the juice to be passed through cloth; which avoid largely the formation of decomposition products of "glucose" during evaporation; which permit crystallisation to take place in a syrup light in colour and free (or almost so) from insoluble matter; and lastly, which enable the molasses or mother-liquor adhering to the mass of crystals (the *massecuite*) dropped from the vacuum pan to be cleanly and easily removed by washing in the centrifugals. When these conditions are fulfilled, a sugar may be obtained of excellent colour, although as a rule one must admit that it is never so faultlessly white as the best crystals refined by the use of animal charcoal.

This improved clarifying effect is produced by the addition of an excess of lime, which is afterwards removed by means either of carbon dioxide (carbonatation), or of sulphur dioxide (sulphitation), a large amount of finely divided precipitate being thus formed in the juice. In this short note, it is impossible to present details of these processes and their several modifications, and space can only be found to describe the chemical effects resulting, so far as these are known.

In regard to the action of carbonatation and sulphitation on the constituents of the juice, it may be pointed out that proteins, nucleins, gums and pectins are partly removed from solution on the addition of the lime, and provided that sufficient of this reagent has been used all the anthocyanin is eliminated also. Altogether a considerably greater precipitation of impurities occurs than that resulting from the small amount of lime used in the defecation process. It is, however, the subsequent formation of the voluminous precipitate of calcium carbonate or calcium sulphite that completes the maximum clarifying effect obtainable; and there seems little doubt that adsorption of the colloidal impurities then occurs. That this is so appears to be supported by the fact that the order of addition of the alkali and acid may be reversed with no

apparent difference in the result; as, for example, when the juice is first sulphited and afterwards limed (which, as a matter of fact, is the usual procedure in some countries).

In a certain important modification of the carbonatation process that is now being used in factories in Java, instead of adding all the lime at once, liming and gassing are carried on almost simultaneously, conditions being so adjusted that only a slightly alkaline reaction is maintained throughout. This variation of de Haan<sup>2</sup> has proved very advantageous in practice for the reasons that the danger of colour formation by the action of an excess of lime on the "glucose" is avoided; that the utilisation of the gas is improved; that the operation is more rapid; that frothing (one of the inconveniences of ordinary carbonatation) does not occur; and, lastly, that less lime is required. But what is of interest to note is that it has been proved that this modification gives the same result in regard to the elimination of impurities as does ordinary carbonatation, in which a large excess of lime is allowed preliminarily to act upon the juice. Since during the de Haan treatment the amount of lime acting upon the impurities is probably little in excess of that used in the defecation process, it is difficult to believe that the elimination of the impurities effected is mainly due to its action. It seems much more reasonable to regard these observations as further evidence that the clarification of cane juice by such processes is largely an adsorption phenomenon rather than a purely chemical process.

In fact several workers, as Deerr, Norris, and Peck<sup>3</sup> have recently emphasised the importance of regarding clarification from the standpoint of colloid chemistry; whilst Geertjens in one of his latest works<sup>4</sup> points out that "the clarification of cane juice by chemicals does not eliminate from the juice any great amount of impurities but it is more the improvement of the physical properties which is effected, as, for example, the removal of the small amount of colloidal matter which hinders the juice from being filtered and keeps afloat the suspended ingredients. . . ."

Furthermore, Zerban,<sup>5</sup> in a publication which has just come to hand, shows by both laboratory and factory experiments that an efficient clarification can be brought about without the use of any chemicals at all; but simply by the use of efficient adsorbents. Kieselguhr and decolorising carbon were the materials used, the procedure being to boil the juice coming from the mills with 0.5 per cent. of the infusorial earth, pass through cloth, heat with about 1 per cent. of high-power decolorising carbon, and again filter. Juice that was brilliant and almost colourless, and that gave an excellent quality and high yield of white sugar, was thus obtained.

This is certainly a result of much importance, which, it may be added, has been confirmed by tests carried out independently in certain factories in Louisiana. Its adoption in practice would depend mainly upon the availability of cheap and efficient adsorbents. There are on the market at the present time several decolorising carbons that are very satisfactory from the point of view of high adsorbing power; but the general opinion of sugar manufacturers and refiners appears to be that these are too costly for extended application. It would certainly be a matter of great interest to discover a means of producing at a low figure a carbon having a high power of adsorption towards the colloidal impurities, e.g., the gums and pectins and colouring matters, present in sugar juices and syrups

<sup>1</sup> U.S. Patent, 1,101,071; this J., 1914, 801.

<sup>2</sup> This J., 1919, 1924.

<sup>3</sup> "Practical White Sugar Manufacture," pp. 52-63.

<sup>4</sup> Louisiana Bulletin, No. 173.

<sup>5</sup> Cf. also German Patent, 332,135; this J., 1920, 668a.

<sup>1</sup> This J., 1920, 667a.

whether this were obtained directly by a simple process of manufacture, or indirectly as a by-product.

Such a carbon, as these experiments of Zerban indicate, or some other, cheap adsorbent, would doubtless find a wide application in the sugar industry. It would probably enable most tropical factories now turning out low-grade "raws" to produce white sugar which would surpass in quality that now obtainable by the carbonation and sulphitation processes. It might ultimately be the means of realising the possibility, which some conceive, of producing directly from cane juice on the plantation a grade of sugar rivalling that now made only in the refinery.

## THE ELECTRIC FURNACE IN THE AMERICAN METAL INDUSTRIES.

In the issue of *Chemical and Metallurgical Engineering* for November 17, 1920, appeared an article by R. M. Keeney on the technology and future possibilities of the electric furnace as applied to the manufacture of iron and steel, ferro-alloys, heat treatment, brass melting, and the smelting of non-ferrous ores. This paper, of which a short abstract is given below, forms a useful complement to the paper on recent developments of the electric furnace in Great Britain, which was read by D. F. Campbell at the annual meeting of this Society in July last (*cf. J.*, 1920, 224 r).

During the last decade great progress has been made both in America and Canada in the application of electric furnaces to the iron and steel industries. Although this expansion was chiefly due to the exigencies of war, it is probable that the electric process will definitely supersede other processes for the production of steel castings and be widely used for the treatment of molten Bessemer and open-hearth steel.

**Pig Iron.**—The production of pig iron from electric smelting furnaces was for the first time proved a commercial proposition by D. A. Lyon at Heroult, California, in 1908. This process has been abandoned, and pig iron is now produced by electric melting of scrap. In Sweden and other countries, however, the development of the ore-smelting process has been steady, and both shaft and open-pit type furnaces have been used. The former resembles Lyon's shaft furnace, which was especially adapted for the production of low carbon and low-silicon iron, whereas with the latter, foundry iron can be produced and coke can be largely substituted for charcoal as a reducing agent. The future development of ore smelting in the United States is doubtful, but there is a possible future for the direct conversion of scrap iron and steel into pig-iron or castings. This synthetic process was largely developed in France, where 220,000 tons of iron was produced during 1916, 1917, and 1918. The electric furnace has also been used for desulphurising cupola metal and for the production of malleable castings.

**Steel.**—The number of furnaces in the United States and Canada has increased since 1907 from 7 to 363, whilst the output of electric steel in the United States for 1918 amounted to 511,364 gross tons. The electric process has found successful application in the manufacture of various alloy and high-speed steels, but when making carbon steel approaching best crucible quality only the purest raw material must be used.

Considerable development in the direction of refining molten metal may be looked for in the future, and already the Illinois Steel Co. has installed ten

30-ton furnaces for a triplex Bessemer open-hearth electric process.

**Electric-steel Castings.**—Electric-steel castings can now be made more cheaply than converter and open-hearth castings, even by firms purchasing their power from outside sources. The rapidity of melting ensures small but frequent supplies of liquid steel, which is very advantageous to foundries. As most foundries operate on a 10-hour day, a load factor of 18 to 30 per cent. only is possible, but where continuous operation is practised the load factor can be raised from 40 to 55 per cent.

**Recent Modifications of Furnace Designs.**—The design of recent types has generally followed the principle of the three-phase Heroult furnace. At the same time there has been a growing tendency towards the use of three-phase current and automatic control, the reduction of the reactance in primary circuits and the elimination of hearth conductors carrying all the current load. The best central station load is obtainable with furnaces operating at a power factor of 0.80 to 0.85.

**Electrical Heat-treatment.**—Considerable progress has been made with electric furnaces for the heat treatment of steel. The heating element consists of granular carbon confined in carborundum fire-sand troughs. The load is easily controlled by using a multi-tap furnace transformer. Single-phase furnaces of 900 k.w. capacity have already been installed, and this system of heating is certain to be greatly developed in spite of its higher cost. In another type of resistance furnace the heating element consists of a nickel-chromium alloy ribbon wound on a suitable framework; such furnaces have been built of 400 k.w. capacity.

**Ferro-alloys.**—In the last ten years the power capacity of ferro-alloy plants in the United States has increased from 20,000 to 200,000 k.v.a., but at the present time probably 75 per cent. of these plants is not operating. Further knowledge of the value of molybdenum as an addition to binary and ternary alloy steels has resulted in very marked increase in production of the ferro-alloy, whereas market conditions during the war were alone responsible for the increased output of ferro-manganese and ferro-chrome. These alloys are best made in three-phase furnaces, whilst the small single-phase furnace of Siemens type is preferred for smelting tungsten, molybdenum, uranium, and vanadium ores.

**Brass.**—The use of electric furnaces for melting non-ferrous metal and alloys has grown remarkably during the past five years. In March, 1920, there were 261 units installed with an estimated total load capacity of 23,000 k.v.a. One-third of this load is taken by resistor furnaces, one half by arc furnaces, and the remainder by induction furnaces of small capacity. For brass-melting the power consumption varies from 250 to 400 k.w.-hrs. per ton. Single-phase furnaces are almost exclusively used.

**Zinc.**—Zinc is not being smelted commercially in the United States, although considerable experimental work has been done and the condenser problem apparently solved. In Sweden 15,000 k.v.a. is being utilised at two plants for ore and dross smelting.

**Future Development.**—The limitation of the electric furnace is primarily defined by the cost of power. For melting steel and non-ferrous metals a price of 1 cwt. per k.w. hour is reasonable, but this would prove altogether prohibitive for the production of ferro-alloys.

Future progress may be expected in the following processes:—(1) Iron castings from scrap or a duplex process of cupola melting with electric furnace refining; (2) steel castings and alloy steels; (3) ferro-alloys; (4) aluminium; (5) brass melting; and (6) electric smelting of non-ferrous ores.

## SOCIETY OF CHEMICAL INDUSTRY.

### JOINT MEETING OF THE SOCIETY WITH THE INSTITUTION OF MECHANICAL ENGINEERS.

A joint meeting of this Society and the Institution of Mechanical Engineers has been arranged, at which Monsieur Paul Kestner, President of the Société de Chimie Industrielle, will read a paper on "The De-gassing and Purification of Boiler Feed Water." All members of the Society will be cordially welcome to the meeting, which will be held at the Institution of Mechanical Engineers, Storey's Gate, Westminster, S.W. 1, on Friday, March 4, at 6 p.m.

### JANUARY MEETING OF COUNCIL.

The monthly meeting of the Council was held on January 14, Sir William J. Pope presiding. Among other matters dealt with the following may be mentioned:—

Arrangements were agreed upon for taking over the Society's share of the estate of the late Dr. Rudolph Messel, and it was resolved to comply with the testator's wish that the fund should be kept separate from the other funds of the Society. As, under the Society's by-laws, the trustees of the fund must not be members of Council, it was agreed to ask Dr. C. C. Carpenter, Mr. E. Grant Hooper, and Prof. G. G. Henderson to act in this capacity.

It was decided to proceed with the printing and publication of the fifth volume of the Society's Annual Reports of the Progress of Applied Chemistry, and that it should be sold to members at the price of 7s. 6d., and to non-members at 15s., plus 9d. postage, in each case. It was further resolved to offer to purchasers of Volume V. a set of the remaining copies of Volumes II., III., and IV. (Volume I. being out of print) at a reduction of 25 per cent. of the total price.

Seventy-nine new members were elected (as compared with 72 in January, 1920), of whom 64 are home members, 8 colonial, and 7 foreign.

The representatives appointed at the request of the National Physical Laboratory to consider its note on "Tests of Graduated Glassware" submitted a report on the subject, which was adopted by the Council, and ordered to be sent to the Director of the Laboratory.

On the nomination of the Committee of the Chemical Engineering Group, Messrs. C. S. Garland, C. L. Goodwin, J. W. Hinchley, W. R. Ormandy, and J. A. Reavell were elected the Society's representatives on the Chemical Engineering Committee of the British Engineering Standards Association.

The Council had before it a report received from the committee of the Nottingham Section on the subject of "The Recent Policy of the Publications Committee." The chairman and hon. secretary of the Section were present and amplified the views set forth in the report. A discussion followed, and the thanks of the meeting were given to the committee and its representatives for having brought forward the matter.

It was resolved that the Society as a whole should participate in the joint meeting to be held with the Institution of Mechanical Engineers on March 4 (*cf. s.*).

### NEWS FROM THE SECTIONS.

#### MANCHESTER.

The fourth meeting of the session was held on January 7 at the Textile Institute, Mr. J. Allan presiding over an attendance of about eighty.

Dr. A. W. Crossley read a paper on "An Industrial Research Association," in which he discussed its objects, its relation to other research

institutions, especially universities, and co-operative laboratories. Dr. Crossley contended that underlying theory was the fundamental object of research work and, applying this contention to the research laboratory established at Didsbury by the Cotton Research Association, said that it had been decided that the work there should not be based on previous industrial experience, but that investigation of the single fibre should be the fundamental object. Dr. Crossley also urged that the publication of all results is of the utmost importance. Among the points brought out in the discussion were the undesirability of reserving accommodation for research at universities to particular firms; that "investigation" was not necessarily "research"; and that much so-called rule-of-thumb work was essentially of a research character.

#### BRISTOL AND SOUTH WALES.

At the meeting held in Bristol on January 6, Mr. E. Walls presiding, Mr. C. E. Boucher read a paper on "Some Aspects of Chemical Work in Pharmacy."

Following a historical introduction, the lecturer discussed the difficulties connected with preparation, standardisation, and preservation, selecting opium as an example. He then outlined the trend of modern pharmacy, and laid particular stress upon the need for duty-free alcohol. The chairman also pressed the latter point, and spoke of the debt that the chemist owes to the pharmacist.

The first meeting of the Section in Swansea was held at the Royal Institution on January 7. Prof. C. M. Thompson, of Cardiff, presided, and members of the South Wales Section of the Institute of Chemistry were present by invitation.

In a paper on "Coal," Mr. C. A. Seyler outlined the development of its classification from the earlier attempts of Regnault, in 1837, to the more scientific methods now adopted, which are based on the relative amounts of carbon and hydrogen in the pure coal substance, and showed how well the results of these agreed with the data derived from the study of the geological and geographical distribution of the South Wales coalfield. He traced by means of charts and maps the gradual changes from the centres of anthracitisation outwards, showing how the physical, chemical, and palaeobotanical characters change concurrently with the changes in carbon and hydrogen contents. The speaker showed slides of present and fossil forms of Lycopods and Sigillarias, compared these with a number of photomicrographs in natural colours, and pointed out the fossil structures therein.

The chairman announced that it was proposed to hold two meetings in Swansea next session, provided papers could be obtained from local members.

#### YORKSHIRE.

The second meeting of the session was held in Leeds on January 17, with Dr. L. L. Lloyd in the chair.

Mr. H. J. Hodsman described and exhibited a new form of apparatus for sodium peroxide fusions, which consists of an elongated steel crucible about 4 in. long by 1 in. in diameter and closed by a well-fitting lid. The crucible is encased in an outer steel tube the cover of which slips on with a bayonet joint. In this apparatus the fusion of organic substances, especially fuels, can be carried out safely and conveniently by simply heating the outer tube in a bunsen flame.

A short paper by Messrs. B. A. Burrell and G. W. Douglas, entitled "The Presence of Nitrates in the Leaves of Trees Growing near a Picric Acid Works," was read by the latter. Damaged and undamaged leaves were examined by extracting with cold water and estimating the nitrate content of



the extract by the colorimetric method, using brucine and oxalic acid. The damaged leaves all showed the presence of nitrate, the amount of which varied from 0.05 to 0.38 per cent. (expressed as nitric acid), whilst the undamaged leaves from the vicinity of the works and also from other districts of Yorkshire showed no trace. It was observed that beech trees were most susceptible to the fumes.

At the conclusion of the meeting a burette-holder, designed by Mr. F. Firth, was exhibited. The burette is held in a clip attached to a jointed arm similar to an ordinary gas bracket, so that it can be pulled forward when required and pushed back against the wall to leave the bench clear when not in use.

#### LIVERPOOL.

On December 17, Mr. P. G. Jackson read a paper on "Boiler Feed Water—from the chemist's point of view," in which he discussed the formation of scale in boilers, corrosion of the iron or steel of which they are constructed, of the brass or gun-metal fittings, overheating, and priming or foaming of the water. The various theories of corrosion, the author believes, are almost all correct, but the electrolytic theory is the only one which, in many cases, can explain why corrosion occurs at a particular point. Oxygen is difficult to remove, and usually must be allowed to remain, although in some cases a small quantity of tannic extract may be added; but as tannic acid is itself corrosive, it should be neutralised with soda, and then used only in small amount as the salt is readily dissociated. Undue aeration of a feed water should be carefully avoided. Corrosion due to oxygen and carbon dioxide may usually be recognised by the presence of gas bubbles on rough surfaces lying more or less horizontally, and where the water circulation is defective; in the latter case the pittings are nearly circular in shape, sharply-defined, and isolated. Much experience is necessary to infer from an analysis whether the magnesium salts present in a water are likely to cause corrosion. Magnesium chloride and nitrate, if present in more than very small quantities, are almost certain to cause it, unless sufficient calcium carbonate is present to neutralise their action. Nitrates, when in concentrated solution, cause trouble in boilers working at, say, 150 lb. per sq. in. pressure; the maximum permissible concentration should not exceed 100 grns. per gall. Iron and ammonium salts are especially deleterious, but, in the author's opinion, sodium carbonate is not corrosive.

After discussing the control of feed-water supplies with and without softening plant, Mr. Jackson described boiler troubles due to "priming," which he classified into "foaming" and "bumping." Foaming occurs readily when either soapy matter or certain colloidal organic substances are present in the water, and is probably due to the presence of large numbers of minute suspended particles acting as nuclei for the formation of steam bubbles and causing too rapid ebullition. Bumping may be prevented or minimised by providing ample steam space, excluding soapy water or saponifiable oils, avoiding excessive concentration of saline constituents, and by excluding finely-divided organic or mineral matter, and oil or other substances which may form a scum on the surface of the water. Overheating of certain parts of the boiler frequently occurs when the metal has become distorted or bulged, and is nearly always due to the presence of oil in the scale; and it is therefore very important to exclude grease from the boilers. Oil should be eliminated by chemical means; ordinary softening treatment will remove it from a hard water (with the precipitate), but for a soft water alumino-ferric should be used.

## MEETINGS OF OTHER SOCIETIES.

### THE CHEMICAL SOCIETY.

In a lecture delivered to the Chemical Society in December last, Sir Robert Robertson, Director of Explosives Research, Research Department, Woolwich, described some recent work carried out there on the "Properties of Explosives."

The lecture embodied an attempt to correlate the explosive properties of simple explosives with their chemical composition and constitution. It was shown that the percentage of oxygen which was required to oxidise the combustible elements in the explosive was intimately related to its properties, and also that constitutional changes, such as the arrangement of nitro-groups in isomeric compounds, were responsible for differences in the effects produced. The properties considered were heat of formation, heat of explosion and gases evolved, chemical stability, sensitiveness to shock, rate of detonation and pressure developed; and in each case the description was prefaced by an account of the most recent methods employed by the Research Department, Woolwich, for determining the values. In many cases quantitative results were obtained which make it possible to measure the properties of explosives in terms of definite physical units, instead of by personal estimates of effect, as hitherto.

The influence of the heat of formation as well as of the completeness of the oxygen balance was illustrated by a comparison of the thermal data given by nitroglycerin, trinitrotoluene and trinitrophenol, reference being made to a new set of values for heat of formation of the most important nitro-derivatives of benzene, toluene, phenol, and methyl-aniline. Values for the heat of explosion, obtained under conditions of standard density of loading of the explosive in its containing envelope, were quoted, and the similarity of trinitrotoluene and trinitrophenol in this respect was traced to the higher heat of formation caused by hydroxylation, as compared with methylation, of benzene.

The factors influencing rate of decomposition were discussed, and examples given of temperature coefficients. Variations in chemical stability were illustrated by a table in which the rate of gas-evolution from a number of explosives was compared at a common temperature, and the influence traced of such factors as the introduction of nitro-groups, of methyl groups, and of the hydroxyl group into the ring, the arrangement of the nitro-groups in the compound, and the physical condition of the explosive undergoing decomposition. The resistance to decomposition by heat was in many cases shown to run parallel with the ease with which the explosive compounds can be hydrolysed. Values for insensitiveness to shock were quoted for a number of explosives, and some effects noted of constitutional arrangements, such as want of symmetry in the disposition of nitro-groups, conducing to greater sensitiveness. The rates of detonation under conditions of high density of loading were given for a few explosives.

The important principle, enunciated by the late Prof. B. Hopkinson, concerning the measurement of the pressure of a blow was illustrated by a description of the work done on this quantitative method as applied to explosives. It consists essentially in the separation of the momentum of the blow into pressure and time, of which the latter is known. Attention was called to the necessity for maintaining constant the density of loading of the explosive and the thickness of its containing envelope. Values illustrating these points, as well as the effect of certain constitutional arrangements on the violence of the detonation were given, as were also indications of the applicability of the method to many problems in explosive research in



which the violence of the blow delivered has to be investigated.

In the second part of the lecture the explosive properties of the Amatols (mixtures of ammonium nitrate and trinitrotoluene) were described. These mixtures were put forward early in 1915 by the Research Department, Woolwich, to meet the shortage of trinitrotoluene, and ultimately were used in enormous quantities for filling shells, bombs, and other warlike appliances.

As Amadol 80:20 has its constituents nearly adjusted for complete combustion, its heat of detonation can be calculated, since the heats of formation of the constituents and final products are known. The values actually obtained for heat of explosion and for gases evolved in the high-explosive calorimetric bomb were shown to be in very fair agreement with the calculated quantities. The chemical stability of these mixtures and the influence on them of certain impurities were discussed, and the values obtained for sensitiveness and rate of detonation were given. The application of the pressure bar to the investigation of these mixtures clearly showed that the ammonium nitrate is not to be looked on as a diluent of the high explosive; it is more than a convenient source of oxygen, being itself an explosive.

The lecture, which contains many numerical values for which the original must be consulted (*Chem. Soc. Trans.*, 119 (1921), p. 1), concluded with a tribute to the enthusiasm of the scientific staff of the Research Department, Woolwich.

At the meeting of the Society held on January 20, Sir J. J. Dobbie announced the deaths of Sir Lazarus Fletcher, head of the mineral department of the British Museum since 1878, of Mr. Charles Simmonds (*cf.* p. 29 a of this issue), and of Mr. S. A. Vasey, who directed the *Lancet* laboratory for twenty-eight years. The vacancies on the Council, to be filled at the annual meeting in March, were also announced.

The list of papers to be communicated contained twenty items, of which the following were read in abstract:—The formation of derivatives of tetrahydronaphthylene from  $\gamma$ -phenyl fatty acids: G. A. R. Kon and A. Stephenson. Quantitative reduction by hydriodic acid of halogenated malonyl derivatives. Pt. I.: The amides of sym. dialkyl and aryl substituted amides of mono- and di-hromomalonic acid: J. V. Backes, R. W. West, and M. A. Whiteley. Dihydroxynaphthaldehydes: G. T. Morgan and D. C. Vining. Researches on residual affinity and co-ordination. Pt. III.: Reactions of selenium and tellurium acetylacetones: G. T. Morgan and H. D. K. Drew. Ortho-chlorodinitrotoluenes. Pt. II.: G. T. Morgan. The constitution of catechin. Pt. III.: Synthesis of o-catechin: M. Nierenstein. Chlorine overvoltages: E. Newbery. The influence of mercury, sulphur, arsenic, and zinc on the catalytic activity of platinum: E. B. Maxted. The conditions underlying the formation of unsaturated and of cyclic compounds from halogenated open-chain derivatives. Pt. I.: Products derived from  $\alpha$ -halogenated glutaric acids: C. K. Ingold.

There will be an informal meeting of the Society on February 3, following an ordinary scientific meeting.

#### ROYAL PHOTOGRAPHIC SOCIETY.

At the meeting held on January 11, arranged by the Scientific and Technical Group, the president announced that the Society's progress medal had been awarded to Mr. F. F. Renwick for researches in the chemistry and physics of photography.

Mr. J. Rheinberg, in discussing "Some New Directions for Photographic Research," dealt

chiefly with the variation of the physical properties of colloids which can be induced by such factors as change of solvent, light action in presence of certain salts, etc. In his own work the most important property which he had controlled in this way was the permeability of colloidal membranes to certain solvents; for example, films of gum arabic or fish glue, sensitised with a ferric salt, became more permeable, on exposure to light, to slightly acidified alcohol, thus allowing the extraction of dye from an underlying collodion film. This property was utilised in the manufacture of a mosaic colour screen and also in preliminary experiments for the production of graticules, not carried to completion because of the discovery of the "grainless photography" method. He referred also to the advantage of using methyl alcohol as a solvent for pyroxylin, thus avoiding the objection to mixed ether and alcohol which by reason of their very different rates of evaporation lead to irregular films on drying.

In the paper on "White Light Development," Mr. B. V. Storr gave a short account of the work of Lüppo-Cramer on the reduction of light-sensitiveness of photographic emulsions produced by certain substances. The desensitising action possessed by developing solutions was found to vary considerably with different developers and to be greatest with amidol in the absence of sulphite; further experiments showed that the active desensitiser is the oxidation product of the developer, and this led to a search among dyes of similar composition to the oxidation products. The most active dyes discovered are those of the safranin class, a reduction of sensitiveness of as much as 600–700 times being obtained with phenosafranin. This property is the basis of the process of white light development, which was later demonstrated by Mr. Storr and Mr. H. Flower by developing Special Rapid and Panchromatic plates in white light, after a preliminary hatching in a 1/5000 dyo solution. The dyo used, of which samples were distributed amongst the members of the Society, was not phenosafranin, but a closely allied substance with some advantages in lesser staining and no tendency to produce veil. Messrs. Ilford, Ltd., hope to place this dye on the market very shortly, thus rendering the process available for general use in this country.

#### INSTITUTION OF PETROLEUM TECHNOLOGISTS.

At the meeting of the above Institution held on January 18, a paper on "The Carbonisation of Lubricating Oils in Internal Combustion Engines," by Mr. F. H. Garner, of the Mellon Institute, Pittsburgh, U.S.A., was read in the author's absence by Dr. W. R. Ormandy.

In this paper the author dealt with the cause of carbonisation of lubricating oils in the cylinders of high-speed internal combustion engines, and showed that the suction stroke of the engine is the most important of the whole cycle from the lubricating standpoint, the leakage of oil from the crank chamber during this stroke being the origin of the carbon deposits formed. The question of tests to determine the tendency of an oil to carbonise under such conditions was fully discussed, and a series of tests was given for carrying out these determinations. The carbonisation is influenced by the amount of asphaltic resins present in the oils. The method of determination of asphaltic resins given by the author was one based on that of Rakusin (*J. Rus. Phy. Chim. Soc.*, 1916, 48, 720) in which the oil is mixed with animal charcoal. The asphaltic resins are adsorbed, and the free or loosely combined oil is then extracted by petroleum spirit, in which the adsorbed resins are insoluble; the latter are then estimated by extraction with

chloroform. Experimental work was carried out on a series of lubricating oils from Texas and Pennsylvania crude petroleum, and it was found that:—(a) Oils of similar distillation range have similar evaporation losses at the same temperature, irrespective of their origin; (b) there is a direct connexion between the evaporation loss and the fire test of an oil; (c) carbonisation, that is percentage of asphaltenes formed, increases on heating much more rapidly than the evaporation loss; (d) in similar oils the resin content influences the extent of carbonisation at high temperatures and also the coke value as determined by Conradson's method. The author stated that Conradson's method is a better criterion of the behaviour of the oil in the engine, as regards the formation of carbon deposits, than the carbonisation tests.

In the ensuing discussion, Mr. L. Archbutt stated that in carrying out carbonisation tests he used a large quantity of oil, heated it for 48 hrs., and then did comparative tests on viscosity and acidity; there was a direct relationship between the results of these tests and the behaviour of the oil in practice. Mr. E. A. Evans expressed the opinion that the question of carbonisation had hitherto been approached from a wrong standpoint, viz., that carbonisation was due to the decomposition of unsaturated hydrocarbons; this assumption was open to question. He was at present investigating the tendency of pure hydrocarbons to carbonise and was obtaining very interesting results. Mr. H. Barringer suggested that the carbonisation might be due to extraneous hydrocarbons present in the oil as now sold; and Mr. G. Pitt that oils should be sold on their carbonisation values. Dr. R. Lessing drew attention to the similarity of the asphaltic bodies to those present in coal tar. Dr. M. B. Blackler stated that he had had considerable experience during the past few years in the treatment of lubricating oils in order to obtain fully saturated medicinal oils, and suggested that the proportion of unsaturated hydrocarbons to saturated hydrocarbons in the oil played a great part in determining the carbonisation values.

Dr. W. R. Ormandy, in reply, mentioned that the Diesel Engine Users' Association had investigated the carbonisation of fuel oils in Diesel engines. In his opinion only coking tests gave reliable indications of the production of carbon in internal combustion engines. The so-called carbon in internal combustion engines was really not carbon, but a highly polymerised hydrocarbon or asphalt.

#### SOCIETY OF GLASS TECHNOLOGY.

The fortieth meeting of the Society of Glass Technology was held at the University, Sheffield, on January 19, Dr. M. W. Travers presiding.

The first paper, which was read by Prof. W. E. S. Turner, was by John Currie on "The Rate of Change of Glass Composition in a Tank Furnace following a Change in the Batch Composition." The tank furnace in question had a deadweight capacity of 106 tons, and was charged every 2 hours during six working days by the addition of 30 cwt. of batch and 4 cwt. of cullet. As it was desired to produce a bottle glass of lower melting point and slower rate of setting, the batch was altered so as to reduce the content of lime and alkali; and it was found that nearly three months elapsed before the composition of the glass produced was nearly identical with that calculated from the new batch.

The following two papers were then presented together by Prof. Turner:—(a) "Problems arising in Tank Furnace Practice through Shortage of Salt Cake": W. E. S. Turner; and (b) "The Relative Advantages and Disadvantages of Limestone, Burnt Lime, and Slaked Lime as Batch Materials": F. W. Hodkin and W. E. S. Turner. (a) Various

substitutes for salt cake had been tried with success; in some cases one-half to two-thirds had been replaced by soda ash. Mr. Gelstharpe, chemist to the Pittsburgh Plate Glass Co., had reported that a good clear glass, having more than a certain lime content, could not be made from a batch in which all the salt cake was replaced by soda ash. Among the advantages claimed for soda ash were:—(1) It was usually purer than salt cake; (2) a considerably greater proportion of its weight remained in the glass; (3) a soda-ash batch melts more readily than a salt-cake batch; (4) soda ash was usually transported more carefully by the railways; and (5) it had been stated that salt cake acted more corrosively on the refractory materials of the furnace. The relative prices of these two materials had, of course, to be considered. (b) The effects of limestone, burnt lime, and slaked lime had been tried in both salt-cake and soda-ash batches and in a batch containing a mixture of these. The form of the lime used had a considerable influence; but other factors were also operative. The higher cost of burnt lime was to some extent offset by the elimination of organic matter, which acted as a reducer in the tank and gave rise to glass of poor colour; *per contra*, it was difficult to handle, and it readily absorbed moisture and carbon dioxide. The use of limestone made the batch more expensive, but the gas it evolved helped to agitate the glass and thus produce greater homogeneity.

#### CORRESPONDENCE.

##### THE PROPOSED NEW REGULATIONS FOR CHEMICAL WORKS.

SIR,—The projected new regulations for chemical works issued by the Home Office (*cf.* J., 1920, 16a) are, as a whole, reasonable and progressive. They are designed to protect the workman whilst following his employment from poisonous and other deleterious substances which advancing science has shown to be inimical to his health, and at the same time to compel employers to maintain such a reasonable standard of "welfare" work and good hygienic conditions of employment as the spirit of the times now regards as essential. Many of the details however, will require adjustment or slight rewording. The regulations regarding rescue appliances, working in offensive places, change of clothing, mess rooms and washing facilities, first-aid and ambulance room, drinking-water supply, are all to the good, and their provision will necessitate little or no expense to modern works, since they are already installed in most of them; and any provisions against poisoning by arseniuretted hydrogen are undoubtedly welcome.

The provision of bath accommodation (Regulation No. 15) has always been a disappointment to the progressive employer, as where installed, it has not been used to any extent by the worker. The provision for flooding and drenching with cold water persons splashed with dangerous corrosive liquids (No. 16. (a) 1.) is new, but would appear to be already covered by the provision of fire water-buckets throughout the works. Facilities for sitting down (No. 21) will, no doubt, meet with considerable opposition, as such provision can be so readily abused.

Regulation No. 29 prescribes that chlorate shall be crystallised, ground, and packed in a room or place set apart for the purpose, the floor of which must be smooth, made of impervious and incombustible material, such as cement, and be washed or sprinkled with water and swept daily. No. 31 (b) provides that all persons employed in such a room or space shall wear boots or overshoes

having no metal in them, and woollen clothing, which shall not be removed from the works for any purpose, and which shall be washed daily after use and thoroughly dried before being worn again. These regulations are very severe and go further than experience has demonstrated to be necessary. The washing or sprinkling with water of the floor is really an additional source of danger—daily sweeping is the greatest safeguard. Regulation No. 5, dealing with precautions to be taken against fires, should be amended so as to enable employers to summon workpeople before magistrates for infringing the regulations.—I am, Sir, etc.,  
T. T. BEST.

Manchester, January 11, 1921.

#### ORIGIN OF THE SOCIETY OF CHEMICAL INDUSTRY.

SIR,—As from time to time erroneous statements are made concerning the locality and the circumstances in which the Society of Chemical Industry was founded, and as a little knowledge of the Society's origin may prove interesting and useful, particularly to the younger generation of chemists, I have pleasure in sending you the following few recollections of those early days.

The Society of Chemical Industry, with its "nearly 6000 members" and the best Chemical Journal in the world, grew indirectly out of the Faraday Club, the members of which were chiefly works chemists in St. Helens. The Faraday Club held its meetings usually at Mrs. Boothby's, in Lime Street, Liverpool, occasionally in Widnes and Warrington, and later on in the North-Western Hotel, Liverpool. The Widnes men apparently grew somewhat jealous of the success of the Faraday Club, and determined to have a bigger society of their own. Mr. John Hargreaves (a younger brother of James Hargreaves of "spinning" fame, and of the Hargreaves-Bird electrolytic soda process) accordingly sent out invitations to a meeting to be presided over by Mr. James Muspratt (a brother of E. K. Muspratt, the fifth President of the Society), at the Officers' Room, Drill Hall, Widnes. The Faraday Club members were invited to this meeting; they attended in force and managed to get the secretary (the late Mr. George E. Davis) and treasurer (myself) and another member appointed on the committee to draft the rules for the new society. Mr. G. E. Davis suggested that the Society should comprise the chemists of Widnes, St. Helens, Runcorn, Cheshire, and S.W. Lancashire. Somebody else—probably Mr. James Muspratt—proposed that the next meeting should be held in Manchester, and that Dr. (the late Sir Henry) Roscoe be invited to preside.

At the Manchester meeting a good deal of discussion took place about the name of the Society, many being in favour of making the engineering side more prominent. Mr. G. E. Davis proposed that the meetings be held alternately in London and the provinces, and I suggested that the Society should be co-extensive with the English-speaking world, with branches or "sections" in the chief chemical centres of England and America. This was carried, and the name finally adopted was "The Society of Chemical Industry." The first general meeting was held in London in 1881, in rooms kindly lent by the Institution of Civil Engineers, and was presided over by Dr. Roscoe.

If I were asked to name those who worked hardest to found the Society, I should certainly say Mr. G. E. Davis, who at the time of the formation of the Faraday Club, in 1875, was chemist at Messrs. Gamble, Gerard's Bridge Alkali Works (afterwards one of H.M. Inspectors of Alkali Works and subsequently editor and proprietor of the *Chemical Trade Journal*). He was the Hon. General Secre-

tary of the Society and a vice-president. I used to chaff him about being like the Irishman, who was "never happy unless he was fighting"; and he certainly "built better than he knew." After him I should name the late Mr. Thomas Tyrer, whom I suggested would make a good Metropolitan Secretary (*cf.* his speech at Leeds, J., 1905, p. 722); and Dr. Roscoe, who became the first President.—I am, Sir, etc.,

DOUGLAS DE L. HERMAN.

Manchester, January 11, 1921.

#### PERSONALIA.

Sir James A. Walker has been nominated by the Council of the Chemical Society to succeed Sir James J. Dobbie as president.

Dr. J. N. Pring, reader in physical chemistry at Manchester University, has been appointed head of the physical chemistry branch of the research department at Woolwich Arsenal.

The Perkin Medal for 1921 has been awarded by the American Section of the Society of Chemical Industry to Dr. W. R. Whitney, director of the research laboratories of the General Electric Co.

The following appointments have been made by the Secretary for Mines:—Chief Inspector of Mines, Mr. T. H. Mottram; Deputy Chief Inspector, Mr. H. Walker; Divisional Inspector of Mines, Mr. J. Masterton; Senior Inspector, Mr. E. H. Fraser.

The following have been elected officers of the Council of the American Chemical Society for 1921: President, Prof. Edgar F. Smith; Directors, Messrs. G. D. Rosengarten and H. P. Talbot; Councillors, Messrs. H. E. Howe, C. L. Alsberg, Allen Rogers, and Lauder W. Jones.

Dr. F. G. Cottrell has resigned the directorship of the Bureau of Mines and has become chairman of the Division of Chemistry and Chemical Technology of the International Research Council. His post at the Bureau of Mines has been filled by the appointment of Mr. H. Foster Bain, mining engineer, and formerly assistant director of the Bureau.

The John Fritz gold medal has been awarded to Sir Robert Hadfield for his invention of manganese steel. The medal was first given in 1902, in honour of Mr. John Fritz, ironmaster, of Bethlehem, Pennsylvania, and the award is made by a committee representing the leading American societies of civil, mechanical, metallurgical, and electrical engineers.

Dr. Henry Peile, late chairman of the Newcastle Section of this Society and chairman of the Newcastle Alloy Co., Ltd., has been appointed a Commander of the Order of the British Empire (C.B.E.) for services rendered in connexion with the war. The same honour has been conferred upon Major Sir Douglas Mawson, Head of the Explosives and Chemical Section, Foreign Trade Department.

We regret to record the death, on January 15, from sudden heart seizure, of Mr. Charles Simmonds, who had served for many years in the Government Laboratory, where he was recently promoted to the grade of Superintending Analyst. Mr. Simmonds was a chemist of wide experience; after serving in the Crown Contracts branch of the Laboratory, he was specially engaged first on steel (in connexion with the old Steel Rails Committee) and then on pottery glazes. More recently he had been working on the industrial uses of alcohol, and his work on this subject was published about a year ago, and reviewed in these columns (*cf.* J., 1920, 46 B).

## NEWS AND NOTES.

## FRANCE.

**Industrial Notes.—Chemical Industry.**—Owing to German competition and a general lull in demand, the situation in the chemical market remains unsatisfactory, and prices in general are falling slowly. Heavy chemicals are not materially affected by the present slump, but there are very few inquiries for special products used in dyeing and bleaching, and for textiles, soap, rubber, and plastic materials. Whereas France formerly obtained heavy chemicals from Germany, she is now sending them to that country and buying finished products instead. French buyers are obtaining indispensable chemicals from Germany rather than England, and the Government has been criticised on this account. The production of calcium carbide is still insufficient, but is to be increased by the erection of a new factory near Ugines, where 32,000 h.-p. is available from the waterfalls of the Doron; the electric steelworks "Paul Girod" is undertaking its establishment.

**Minerals and Fertilisers.**—The exportation of bauxite, hitherto prohibited, is now allowed to a limited extent to England and America, on payment of a duty of 25 per cent. *ad valorem*, the object being to improve the rate of exchange.

Florida phosphate has reappeared on the French market, doubtless owing to the fall in freights. In some quarters it is held very strongly that such foreign material ought not to be imported, and that France should derive supplies from her own North African colonies; yet some superphosphate works have had to close down from shortage of raw material. The tardy development of the phosphate beds in Algeria and Tunisia is variously ascribed to shortage of freight, of labour, and lack of railway material, but the true cause is probably lack of organisation at the mines. An agreement has been made to supply Italy with 500,000 tons of phosphate yearly in return for 8000 t. of pyrites and a certain amount of labour.

Opinion in Alsace is against the Government scheme for working the potash deposits, by which exploitation would be carried out by four companies; it is held that the State should do the work, that all the employees engaged in the industry should have a share in the profits, and that farmers should be granted priority in regard to supply and favoured terms as regards prices. Such concentration, it is thought, would be the best means of fighting German competition, which is expected to become very severe.

It is reported that petroleum fields have been discovered close to the French frontier in Spanish Morocco.

**Metallurgy.**—The general situation in 1920 was little better than that during the last six months of 1919; the same factors—shortage and high price of coal, transport difficulties, and high railway rates—continued to impede production, and hence the rolling mills were unable to quote definite prices or dates of delivery. The steelworks outside the invaded area have attained 70 per cent. of their pre-war monthly output, but the restored Lorraine works only 41.1 per cent. The total production of steel during the first six months of 1920 was 1,204,701 tons, and that of pig-iron 1,358,856 t., including 1,325,937 t. made in the blast furnace and 32,919 t. in the electric furnace.

The Comité des Forges de France, on the occasion of its fiftieth anniversary, has published an interesting work on "La Sidérurgie Française," in which, among other points, the prospects of the French iron and steel industry are discussed. It is estimated that when the devastated works of northern and eastern France are in full working

order the French production, including that of the Saar district, will be 11 million tons of pig-iron and 10.4 million tons of steel, or, in the event of the Saar district being restored to Germany in fifteen years' time, 9.4 million tons of pig-iron and 8.1 million tons of steel. In 1913 production and consumption were approximately equal, viz., 5.2 million tons of pig-iron and 4.8 million tons of steel. As Lorraine and the Saar do not absorb their own production, it follows that after pre-war conditions have been established and the wants of the devastated areas fully met, there will be a good surplus available for exportation.

At the moment German competition in the metallurgical market is very active, and quotations from Germany are frequently 50—70 per cent. below the French. The reduction in the price of metallurgical coke from 175 to 135 francs per ton will enable French producers to bring their prices more into line with those of their competitors, although the latter have a decided advantage as regards price of coal and labour.

## BRITISH INDIA.

**Fermentation Industries.**—In India, as in other countries, attention is being directed to the possibility of producing alcohol for power purposes, and the Bombay Government proposes to erect an experimental distillery. As it is, of course, undesirable to use foodstuffs for this purpose, the choice of raw materials is considerably restricted. Alcohol for burning and for industrial purposes has been manufactured on a considerable scale from the *mahua* (or *mawa*) flower, which contains a large proportion of levulose, but the supply of this material is not very great, and the cost of collection would become serious if the scale were enlarged. The possibility is being considered of obtaining alcohol from the rice straw and husk which are available in large quantities in Burma, and especially at Rangoon, where the principal rice mills are situated.

At Nasik, in Western India, a Government factory of considerable size has been erected for the manufacture of butyl alcohol and acetone by the Weizmann process. Nasik was selected as the locality because it is a good centre for *mahua* in spite of the fact that *mahua* had never been used for this process. It has now been found that the most advantageous raw material is low-grade rice, unfit for human consumption, from the Rangoon rice mills. This is now being utilised with technical success, but it is doubtful whether a commercial success can be achieved because the cost of the factory was very high.

## CANADA.

**Glass Production.**—The Canadian Libby Owens Sheet Glass Co., Ltd., is about to erect a plant at Hamilton, Ontario, with a capacity equivalent to 65 per cent. of the total Canadian importations of sheet glass. All the raw materials will be of Canadian origin, e.g., soda ash from Amherstburg, salt cake from Hamilton, and silica from Cayuga, etc. It is proposed to erect another plant to supply the Western Provinces. (*Ch. of Comm. J.*, Dec. 31, 1920.)

**Maple Sugar.**—The maple sugar industry has expanded considerably during recent years, particularly in Quebec, where the production in 1920 was about 13,393 long tons, as against 4911 t. in 1911. Similarly, the output in Ontario was 3125 t., compared with an average for previous years of 2232 t.; in Nova Scotia and New Brunswick, the only other provinces producing maple sugar, the production was, roughly, 223 t. The exports of maple sugar also show an increase, the total in 1919-20 being 1788 t., which compares with 653 t. in 1914-15. (*Official.*)

**Discovery of Silver.**—It is reported that extensive deposits of silver have been discovered near Keno Hill, in the Yukon territory, and it is hoped that their exploitation will compensate for the declining production of gold.—(*Official.*)

#### UNITED STATES.

**The Dye Industry and the Longworth Bill.**—Dye manufacturers in the United States have had to face a similar crisis to that which recently threatened British manufacturers, and they are looking forward to the speedy enactment of the Longworth Bill, which passed the House of Representatives in September, 1919, but has since been held up by the Senate. The Bill provides that only such dyes shall be imported as are not made in the country 'in adequate quantity, of good quality, and at reasonable prices.' It was primarily intended that the Act should be administered through a joint committee of producers and consumers, but it was subsequently decided to entrust this work to the Tariff Commission. After conferences between this Commission and the Senate Committee on Finance, it was agreed to publish a list of dyes which might be imported in order to avoid inconvenience to the consumer. The Senate Committee reduced the higher tariff rates proposed in the Bill, thus making the embargo less rigorous, and in this amended form it recommended the Senate to adopt the measure. However, when the Bill came before the Senate last spring it was "talked out" by its opponents.

The American Chemical Society has issued a bulletin urging strongly that the Bill be considered and passed without delay. Among the arguments advanced are, (1) a self-contained and independent dye industry is a strong weapon for national defence; (2) large quantities of German dyes are now coming on the American markets; (3) the development of the home industry is being crippled by the uncertainty of legislation; projected extensions of plant have been postponed, research staffs reduced, and much of the capital invested in laboratory plants and apparatus (\$100,000,000) remains unremunerative. Further procrastination is condemned not only by the large dye firms, but also by the small manufacturers, on behalf of whom it was declared in the Senate that they would be the first to go under in the price war which would ensue if adequate legislation were not enacted. The home dye industry is now able to meet 85 per cent. of the needs of the country; the remaining 15 per cent. includes the special dyes which only a fully developed industry can be expected to produce.

**The Dye Industry in 1919-20.**—According to a report issued by the Bureau of Foreign and Domestic Commerce, the United States now possesses a dye industry capable of meeting all home demands. The tendency to consolidate American dye enterprises has not only increased consumption but also developed foreign trade. The value of the aniline dyes exported during the year ended June 30, 1920, viz., \$17,130,397, represented an increase of 68 per cent. over that of the previous year. Imports of alizarin and alizarin dyes amounted to 115.1 tons (\$103,266), compared with 5.3 t., valued at \$13,286, in 1918-19, practically all of which was derived from the United Kingdom; it is reported, however, that one of the largest American makers can now supply certain alizarin colours. Imports of other finished coal-tar dyes rose from 863.4 t. (\$2,753,116) to 1224.1 t. (\$3,775,216), or an increase of 41 per cent. Switzerland has replaced Germany as the chief source of imported coal-tar colours; the United Kingdom supplied 275.3 t., worth \$609,238, in 1919, and 249.1 t., worth \$567,258, in 1920. Imports of natural indigo declined from 44.6 t. (\$1,242,855) in 1919 to 56.5 t. (\$165,332) in 1920, but that of synthetic indigo increased from

265.2 t. (\$308,582) to 452.7 t. (\$530,285); it is considered unlikely that natural indigo will be able to compete with the synthetic product. Owing to the need of statistical data concerning the foreign trade in coal-tar colours, a new import classification has been drawn up which takes effect as from January 1, 1921.—(*Bd. of Trade J., Dec. 9, 1920.*)

**Sodium Compounds in 1919.**—The total production of manufactured sodium compounds was 9,393,749 short tons, valued at \$121,204,219 in 1919, compared with 10,199,493 t., worth \$142,788,535, in 1918. Of sodium salts derived from natural sources 7,093,138 t. was produced in 1919, against 7,262,797 t. in the previous year. Imported sodium salts showed a very great decrease, viz., 522,356 tons, compared with 2,111,725 t. in 1918, but imports of sodium cyanide, ferrocyanide, and sulphide notably increased. The exports, valued at \$18,545,420, consisted chiefly of common salt (119,416 t.), caustic soda (82,118 t.), soda ash (50,481 t.), washing soda (5,563 t.), and sodium silicate (12,150 t.); those of soda ash decreased greatly, but that of caustic soda increased. The substitution of sodium compounds for potassium compounds, initiated during the war, continued during 1919.

#### Production of sodium and sodium compounds in the United States in 1919.\*

	Quantity Short tons
Sodium acetate .. .. .	2425
Sodium benzoate .. .. .	126
Sodium bicarbonate .. .. .	134,962
Sodium bichromate .. .. .	26,523
Sodium bisulphite and sulphite .. .. .	11,846
Sodium bromide .. .. .	499
Sodium carbonate—	
Soda ash .. .. .	981,054
Monohydrate and sesquicarbonate .. .. .	38,795
Sal soda .. .. .	80,090
Sodium chlorate and sodium perchlorate .. .. .	1210
Sodium chloride—	
Salt in brine .. .. .	2,800,000
Rock salt .. .. .	1,637,300
Evaporated salt .. .. .	2,618,200
Sodium citrate, tartrate, bitartrate .. .. .	33
Sodium cyanide, peroxide, iodate .. .. .	9148
Sodium ferrocyanide .. .. .	8437
Sodium fluoride, acid sodium fluoride, sodium fluosilicate .. .. .	811
Sodium hydroxide (caustic soda) .. .. .	855,466
Sodium iodide .. .. .	12
Sodium nitrate (refined) .. .. .	8040
Sodium nitrite .. .. .	676
Sodium phosphate (including all sodium phosphates) .. .. .	14,760
Sodium silicate .. .. .	800,138
Sodium sulphate—	
Salt cake .. .. .	134,685
Glauber's salt .. .. .	42,087
Sulphate .. .. .	83,402
Sodium sulphide .. .. .	45,448
Sodium tetraborate (borax) .. .. .	28,518
Sodium thiosulphate (hyposulphite) .. .. .	52,212
Miscellaneous sodium compounds .. .. .	841
Total .. .. .	9,393,749

\* For production in 1917 and 1918 cf. J., 1920, 8a.

In the above returns the production of sodium cyanide is combined with that of peroxide and iodate in order not to reveal the production of individual firms. Sodium cyanide is now being supplanted by a product, presumably calcium cyanide, made by heating calcium cyanamide with salt to a high temperature and quickly cooling the melt. The reaction is conducted continuously in electric furnaces with conducting hearth and a single suspended electrode; it is used directly in the extraction of gold and silver and for making sodium ferrocyanide and hydrocyanic acid.

The production of caustic soda given in the table does not include that made in soap works. Many pulp and paper mills also make caustic soda for their own use.—(*U.S. Geol. Surv., Nov. 16, 1920.*)

**Foodstuff Preservation with Condiments.**—The Glass Container Association of America has published a monograph by K. G. Bitting on the "Effect of Certain Agents on the Development of Some Moulds," in which the observations of some fifteen

years are recorded. The work has special reference to the use of spices or other condiments as preservatives for foodstuffs, and in addition to containing numerous tables, it is profusely illustrated with photomicrographs showing the effect of many materials upon typical moulds.

**New Use for Mercury.**—The utilisation of mercury vapour for the production of power is being investigated in the United States, the object being to increase the range of temperature through which the heat from the fuel may be converted into power. The mercury is vaporised in a special boiler at about 10 lb. gauge pressure and used to drive a turbine. The exhaust from this turbine is condensed in a form of surface condenser, which produces steam from the cooling water, and the steam is utilised to drive another turbine, the condensed steam in this case being used as cooling water for the mercury condenser. It is claimed that the use of this device in a modern power station would increase the capacity by about 60 per cent., while increasing the fuel consumption by only 15 per cent., and trials with over a 1000 k.w. on the mercury turbine are stated to have fully realised the economies predicted.—(*Power*, Oct. 27, 1920.)

#### SOUTH AFRICA.

**Mineral Output in 1919.**—The Annual Report of the Secretary for Mines and Industries, 1919, gives the mineral production for that year and for 1918 as follows:—

	1919. Tons.	1918. Tons.
Coal .. .. .	10,266,135	9,377,325
Copper .. .. .	4,885,319	6,874,359
Tin .. .. .	1,630,168	2,206,042
Antimony .. .. .	32,000	99,055
Arsenic .. .. .	8,120	17,785
Lead .. .. .	754,000	161,600
Manganese .. .. .	165,000	544,250
Tungsten .. .. .	4,469	18,880
Asbestos .. .. .	3,932,077	3,674,053
Corundum .. .. .	178,567	3,875,526
Graphite .. .. .	85,511	78,974
Iron pyrites .. .. .	5,532,234	4,630,000
Magnetite .. .. .	1,024,000	832,500
Mica .. .. .	2,951	5,089
Mineral paints .. .. .	239,809	735,556
Soda .. .. .	52,050	794,340
Talc .. .. .	756,750	670,250
Gold .. .. .	8,331,451.142	8,418,379.029
Silver .. .. .	891,303.635	877,522.147
Diamonds .. .. .	2,588,017.06	2,537,360.16
	Carats.	Carats.

\*Mostly contained in other ores.

The low-grade gold mines on the Rand are stated to be in a serious position, and labour troubles are reducing the output of the more productive properties. Attention is now being concentrated on base metals and minerals, such as iron ores, asbestos, corundum, mica, and, to a lesser extent, salt, lime, gypsum, soda, magnesite, and graphite.—(*Bd. of Trade J.*, Jan. 6, 1921.)

#### JAPAN.

**The Dye Industry.**—The U.S. Bureau of Foreign and Domestic Commerce has received information from Tokyo to the effect that the Japanese dye industry cannot compete with German or American manufacturers, even with the help of the new import duty of 35 per cent., owing to lack of technical skill in their production and of a home supply of intermediates.—(*U.S. Com. Rep.*, Nov. 23, 1920.)

**Imports of Dyestuffs in 1920.**—Since the end of the war the new Japanese dyestuffs industry has suffered to such an extent from the revival of European competition, that in March, 1920, 72 works, or 65 per cent. of the whole industry, had to close down. Concurrently the importation of dyes rose from 130 tons in January to 262 t. in May, and fell during the industrial crisis in June to 165 t., the total for the six months ended June, 1920, being 1025 t., as against 223 t. in the same

period of 1916. The imports of European and American dyestuffs have steadily increased since 1916, although there was a large decrease in 1917. The average price of dyestuffs in Japan in 1920 was 4.75 yen per kin (98.84d. per 1.32 lb. at par value).—(*Z. angew. Chem.*, Dec. 24, 1920.)

#### AUSTRALIA.

**The Iron-Ore Deposits at Yampi Sound, Western Australia.**—The Queensland Minister for Mines announces that his Government has decided to exercise the option of purchase which it holds over the vast iron-ore deposits in Cockatoo Island, Yampi Sound (*cf. J.*, 1920, 218 a), with a view to establishing State iron and steel works in Queensland. The Minister stated that the opinion of the manager of Messrs. Armstrong, Whitworth and Co. (Mr. Cooper) was that the manufacture of steel from ore as rich as that at Yampi would be extremely easy.—(*Official*.)

**Gold Mining, etc., in Western Australia.**—The Imperial Mineral Resources Bureau reports a noteworthy development on the abandoned Golden Dream lease, about one mile north of Hannam's Reward mine, Western Australia. An auriferous lode has been struck and driven on for 60 ft. over an average width of 2 ft. 6 in., and a trial crushing gave 95 oz. of gold from 88 tons of rock, with 5 dwt. per ton in the sands.

The production of gold at State Batteries since the date of inception to August 31, 1920, has now reached a total value of £5,230,839, made up as follows:—Tons of ore milled, 1,266,431.69; production by amalgamation £4,379,655.52; by sands treatment £634,354.64; by slimes treatment £207,475.47; and by residues £9353.37. In addition, the State tin-treatment plants have produced by black tin £92,120.93, and by residues treatment £572.22, making a total mineral value from all sources of £5,323,532.25.

#### GENERAL.

**Research in the Brewing Industry.**—An editorial in the current issue of the *Journal of the Institute of Brewing* expresses regret that the activity in brewing research which marked the halcyon days of O'Sullivan, Horace and Adrian Brown, Hearn, and Morris, has not been sustained in recent years, and ascribes the lack of progress mainly to the restrictive influence of State supervision. The fact that the brewing and distilling industries are valuable sources of revenue to the State hampers initiative, stereotypes processes, and fosters empiricism at the expense of scientific advance. The Amylo process of alcohol production, for example, is not permitted in this country because of a statutory regulation which prescribes that the strength of distillers' worts before fermentation must be ascertained by the saccharometer, a method which is not practicable in the process named.

In order to promote and develop organised research, the Institute of Brewing has drawn up a comprehensive scheme, and has succeeded in raising a capital sum which will allow of £5000 per annum being devoted to this purpose. Investigations on the breeding and cultivation of hops have been undertaken at the South-Eastern Agricultural College at Wye and at the Malling Fruit Research Station, whilst experiments on the drying of hops and on the effect of manurial treatment on the growth have been made possible by the generosity of private donors. Concomitantly with these searches Prof. F. L. Pyman, at the Manchester College of Technology, is to investigate those constituents of the hop which determine its preservative and antiseptic properties; and work on bark is also to be taken in hand. Arrangements have been made for the investigation of the suitability of various species of oak for cask-making (supply



from Memel being no longer available) at the Imperial College, South Kensington, where Prof. F. B. Schryver and Prof. P. Groom will attack the problem from the chemical and botanical sides respectively. The Institute has also made due provision for the encouragement of research by individual private workers.

**The Science of Nutrition.**—The Huxley Lecture delivered by Prof. Gowland Hopkins at Charing Cross Hospital Medical School on November 24, 1920, was devoted to a review of the striking modifications which the theories of animal nutrition have undergone during the last twenty years.

At the close of last century the problems of human nutrition were being more and more regarded from the standpoint of energetics, and dietaries were usually considered solely on the basis of nitrogen content and calorific value. Although it is true that many of the foundations of the science of nutrition were laid by investigators who regarded the problems they attacked in this manner, there was a tendency on the part of many to carry this conception too far and to neglect the influence of other factors. Rubner, to whom we owe so much of our knowledge of the energy requirements of the animal body, was led away in this manner, and gradually developed a theory which disregarded, in one sense, the significance of the chemical composition of the diet and the material changes which occur in the body, and gave sole prominence to the calorific values of the constituent foodstuffs. The law of Isodynamic Equivalence, propounded by Rubner, states that the living tissues demand energy alone, and from his works one infers that considerations of energy exchange are alone adequate to explain such phenomena as growth, body size, and length of life. It does not require a very deep examination of his views to show how misleading they may be, and, indeed, have been. If fat and carbohydrate are interchangeable as sources of energy for the animal tissues, as follows from Rubner's law, it should be possible to derive the whole energy supply from a dietary of fat. Such an extreme case is one of the many examples where the theory fails, for it is well known that such a diet causes a definite breakdown in the internal metabolism of the body with the production of the abnormal condition known as ketosis.

Closely following the theories of Rubner we have the system of nutrition introduced by Prof. von Pirquet, of Vienna, who evaluates dietaries in terms of a unit called the "Nem," which is the energy supplied by the combustion of the constituents of 1 c.c. of milk. Here we have a practical system in which the chemical details of the diet are disregarded, and yet one which is being employed on a large scale to-day in Central Europe in the organisation of the relief feeding of nearly a quarter of a million children in Vienna on behalf of the Red Cross organisation. Such systems as these are now being vigorously attacked by investigators, mainly of the English-speaking races, who hold that the chemical composition of the diet is as important as the calorific value. It is now established that the character of the protein supply is all important to the animal body, and that the biological value of the proteins may show wide variations, which can be traced to their content of certain indispensable amino-acids. Further, the discovery of the vitamins and recent work in this wonderfully interesting field of research have demonstrated that nutrition and health may fail even when an animal is consuming a dietary adequate as regards protein, calories, and fats unless ample supplies of these unidentified accessory factors are present. Rubner and his followers would regard energy supply as the one essential limiting factor in nutrition, but it must

now be recognised that there are many factors which may act in this manner. In this country and America there remain few who do not acknowledge this to be true, but in Central Europe the older theories still hold their ground. They are, however, being attacked in a manner that promises to demonstrate their inaccuracy. Dr. Harriette Chick and Dr. Elsie Dalyell, under the auspices of the Lister Institute and the Medical Research Council, are now in Vienna, where in two adjacent wards of the hospital of von Pirquet the new theories are being tested against the "Nem" system under rigidly controlled parallel conditions. As Prof. Hopkins remarked, "the enterprise is unique in its occasion, in its international character and in the high qualifications of those who are conducting it." One cannot help foreseeing that this experiment will show how important to practical medicine are recent advances in the science of nutrition.

**The Sweetness of Saccharin.**—The sweetening powers of saccharin and of its hydrated sodium salt are conventionally accepted at the fixed values of 550 and 420, respectively, compared with that of refined beet sugar, but recent investigations by Prof. Th. Paul have shown that the sweetness is dependent upon the concentration of the saccharin solution tested; it increases at a slower rate than the concentration. Thus the sweetness of the sodium salt (in concentrations corresponding to 2–10 per cent. sugar solution) was found to vary between 200–700 times that of sugar, and not to be 420 times, as generally accepted. It was also found that the sweetness of an aqueous solution containing saccharin and other artificial sweetening agents was approximately equal to the sum of the sweetening powers of the constituents. These results should assist those concerned in estimating the amount of sweetening agent required for any particular foodstuff.—(*Schweiz. Chem.-Z.*, Nov. 30, 1920.)

**Proposed Drug Monopoly in Sweden.**—There are grounds for believing that the Swedish Government intends to create a monopoly in the manufacture of pharmaceutical preparations, and possibly also to nationalise the whole industry. According to the present law, the manufacture of medicinal preparations by druggists is restricted to that of certain articles, but in recent years almost all descriptions have been manufactured in a large number of private laboratories, and effective control has been completely lacking. It is provisionally planned that a new central institution shall take over all existing privately-owned works, but that individual pharmacists shall be free to manufacture for themselves or to purchase from the institution. Owing, however, to the superior economy of large-scale production, it is considered certain that in nearly every case druggists will be driven to adopt the latter course. The institution mentioned will be established by a new subsidiary company of the Wine and Spirit Central Organisation in Stockholm, which will have a capital of 5 million kronor (£281,250 at par), and every Swedish pharmacist will be summoned to take shares, the Central Organisation retaining a majority. The works of the A.B. Astras Fabriker in Söderfälje will then be acquired and manufacture proceeded with. Finally, an opportunity will be given to the State to acquire a controlling interest in the undertaking.—(*Chem. Ind.*, Nov. 17, 1920.)

**The Porcelain Industry in Norway.**—All porcelain used in Norway for technical purposes formerly came from Germany, but war conditions gave the spur to the development of a home industry. The Norsk Teknisk Porzellanfabrik, Frederikstad, is now in a position to supply technical porcelain ware in great variety, more particularly electrical insulating ware, and to hold its own against foreign competition. The company was formed during the



war, began to produce regularly in November, 1919, and now commands a capital of 1.1 million kronor (about £62,000 at par). Norwegian feldspar and quartz, and German kaolin are the raw materials employed, and as the factory has ready access to the harbour, transport arrangements are very favourable. It is candidly admitted in Norway that the installation and manufacture would have been impossible but for the help of German technologists and workpeople, who have been employed since the beginning. German labour is used for all difficult work, and the technical director is a German who for a long time held a prominent position in a leading German factory.—(*Z. angew. Chem.*, Dec. 10, 1920.)

**Cost of a German Degree Course in Chemistry.**—The present cost of a four-year course (eight semesters) in chemistry is estimated to be about 6000 marks (about £300 at par, now £22—27), exclusive of the cost of materials required for the "Doktorarbeit," and of the fees payable upon graduation (500 mk.). The cost is distributed as follows:—First semester, lectures 250 mk., books 200 mk.; second semester, lectures 250 mk., books 100 mk.; third semester, qualitative analysis, practical physics, etc., 400 mk., materials 450 mk.; fourth semester, qualitative and quantitative analysis, practical physics, etc., 400 mk., books 100 mk., materials 400 mk.; fifth semester, quantitative analysis, special and general lectures, organic preparations, 350 mk., books 150 mk., materials 700 mk.; sixth semester, organic chemistry 350 mk., books 100 mk., materials 700 mk.; seventh semester, "Doktorarbeit," roughly 300 mk.; eighth semester, 300 mk. Thus the cost of University fees is about 2600 mk., books 650 mk., and chemicals, apparatus, etc., 2250 mk.—(*Chem.-Z.*, Nov. 13, 1920.)

**Chromium Ore in Russia.**—The chief deposits of chromium ore in Russia are on the eastern slopes of the Ural mountains, and although little is known about them, their large number and their proximity to metallurgical centres should lead to development when conditions permit. There are no data available concerning production, but it is known that ore has been obtained from 20 or 30 small mines by means of primitive methods. Chromium ore is stated to occur in abundance on the north-east shores of Lake Koktcha, Government of Erivan, Transcaucasia, and also along the Gazimur River, near the Kuluminsk silver-lead mines in Transbaikalia.—(*U.S. Com. Rep.*, Sept. 20, 1920.)

**Possible Sources of Wood-Pulp in Chile.**—The utilisation of the timber resources of Chile should receive increased attention as the more accessible wood-pulp material ceases to be available. Analysis of certain woods and reeds by the soda process has given the following results:—*Olivillo* gave 38 per cent. of a dark pulp, difficult to bleach, but quite suitable in its unbleached state for the manufacture of wrappings. The average length of the fibre was 2.15 mm., and a sheet 0.0044 in. thick showed a bursting strength of 23 lb. per sq. in. *Coigue* yielded 42 per cent. of a pulp readily bleached; the fibres were short (0.86 mm.), but in conjunction with sulphite pulp it could be used for the production of printings. *Lingue* closely resembles *Coigue*, but gives slightly longer fibres (1.11 mm.). *Quilla*.—This reed occurs abundantly and can be harvested annually; it gave a pinkish-coloured pulp with an average length of fibre of 2.22 mm., and could be used unbleached for newsprint. The bleached pulp could be used without the addition of sulphite pulp for the production of light printings. There are other woods said to be equally well or better suited for pulp-making, such as the Araucanian pine, which grows abundantly and rapidly and gives a dark-coloured pulp, with an average fibre length of 2.36 mm.—(*U.S. Com. Rep.*, Oct. 23, 1920.)

## REPORT.

REPORT ON THE TRADE, INDUSTRY AND FINANCE OF MOROCCO. *Department of Overseas Trade.* London: H.M. Stationery Office. 1920. [Cmd. 975. 1s.]

The French Protectorate of Morocco has numerous natural resources, many of which are either unutilised or are being developed on an experimental scale. Industry is now developing freely under conditions closely resembling those which existed before the war, and, with the exception of certain agricultural produce, the export trade is unfettered by restrictions. The mineral resources are still undeveloped, but important oil-fields and phosphate beds will probably be exploited in the near future. Industrial development, hitherto very backward, has made rapid strides since 1914; factories have been erected for the manufacture or preparation of cement, lime, palmetto-palm fibre, meat and other foods, as well as flax-spinning and olive-oil mills, electrical power stations, and a large modern slaughter-house with by-products factory. Progress is reported in the cultivation of flax, and prospects of producing spun flax and hemp are considered very hopeful; however, with the exception of wool and the palmetto palm, raw materials are not abundant. There has been a great shortage of fuel, and skilled labour is scarce. Exports in 1919 included:—Linseed, 15,800 tons (£840,952), and hides and skins, 3248 t. (£782,783). Imports consisted chiefly of building materials (e.g., cement 3794 t.), cotton textiles, sugar (43,884 t.), candles (3579 t.), soap, kerosene, and gasoline; there is a large demand for cement, glassware, gasoline, kerosene, and coal.

The resources of the Tangiers Zone are confined to agricultural products. There are no railways in the area, but work has been started on a line between Tangiers and Fez. Exports in 1919 were valued at £289,669, including beeswax (£4310), and skins and hides (£192,117); imports were valued at £1,563,650. Great Britain has retained its pre-war predominance in the import trade of many articles, e.g., candles and soap, and has taken the place of France as the chief source of supply of paints, but has lost practically the whole of the trade in vegetable oil to Spain. Good openings are available for British exporters of building materials and window glass, cement, fertilisers, glass and china.

The agricultural prospects of the Spanish Zone of Morocco are good; the palmetto palm is very abundant, and its fibre and leaves are used by the natives for making baskets, mats, etc.; extensive cork oak forests exist in the unpacified areas, and a great future is anticipated for the cultivation of olives. The occurrence of ores of iron, copper, zinc, lead, silver, and other minerals is known with certainty, but adequate surveys of their nature and extent have hitherto been impossible owing to the unsettled state of the country. Three iron-ore and one lead-and-zinc mine are being worked in the Melilla district, the most important of which is the iron-ore mine of the Espanola Minas del Riff company, which has a monthly output of 15,000 to 20,000 tons assaying Fe 63%, SiO<sub>2</sub> 5%, and a low phosphorus content. An output of 50,000 tons a month is considered possible. From the Navarette mine of the Solazar company, 61,000 t. of ore was shipped to the United Kingdom in 1918, and the annual output is expected to reach 150,000 to 200,000 t.; the ore averages 57.5 per cent. of iron, but is rather high in phosphorus. The Alicante company has produced about 800,000 t. of manganiferous iron ore since 1915; its deposit is superficial, but is estimated to contain up to one million tons of ore with about 55 per cent. of iron. The Afra leuc

and zinc mine is worked by the Norte Africano company, which has produced a maximum yearly output of 1500 t. of galena. This mineral is of high grade, but non-argentiferous; the zinc ore is calamine with about 32 per cent. of zinc. A new washing and concentrating plant of large capacity has been installed at the mine by a German firm, but has not yet been used. Numerous deposits of iron ore are said to occur in the unpacified zone, and other reported occurrences are lead, copper, antimony, and petroleum. Both the export and the import trade are almost entirely in Spanish hands; in the former England is badly handicapped by absence of direct shipping communications. The exports from Tetuan in 1918 included hides and skins, tanned hides, tamarisk gail, fullers' earth, linseed, virgin wax; and among the imports of British origin in 1918 were ultramarines, perfumery, soap, candles, linoleum; and in 1917 refined sugar, linseed oil, sarsaparilla, varnishes, ochres and ultramarines, perfumery, soap, candles, glass, and matches.

The report contains a large sketch map of the country, appendices giving statistics of trade, revenue, prices, etc., and reports on minerals submitted by the Imperial Institute to the Department of Overseas Trade (cf. J., 1920, 382 a).

## OFFICIAL TRADE INTELLIGENCE.

(From the Board of Trade Journal for January 13 and 20.)

### OPENINGS FOR BRITISH TRADE.

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W. 1, from firms, agents, or individuals who desire to represent the U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number:—

Locality of Firm or Agent.	Materials.	Reference number.
Australia ..	Perfumery .. .. .	80
" ..	Paint (oxide of iron) .. .. .	*412/20/ 7,392
British India ..	Glass, crockery, porcelain, soap, perfumery .. .. .	83
Canada ..	Galalith wares .. .. .	85
" ..	Paper mill materials .. .. .	91
" ..	Soap, toilet preparations .. .. .	42
" ..	Heavy chemicals, dyes .. .. .	43
Egypt ..	Asbestos card .. .. .	†
" ..	Oil, tyres .. .. .	97
New Zealand ..	Oils, colours, varnish .. .. .	45
" ..	Chemicals, paper .. .. .	46
South Africa ..	Mining chemicals .. .. .	51
" ..	Toilet soap .. .. .	95
Belgium ..	White lead in oil, powdered antimony .. .. .	53
" ..	Sole leather .. .. .	99
" ..	Industrial oils .. .. .	100
" ..	Industrial and pharmaceutical chemicals .. .. .	102
Denmark ..	Chemicals, lubricating oils .. .. .	60
Germany ..	Fats, oils, grease, tallow .. .. .	63
Hungary ..	Chemicals, drugs, indigo, soap, oils, sugar, paper .. .. .	108
Norway ..	Tinplate .. .. .	110
Spain ..	Iron .. .. .	113
Switzerland ..	Colours, paper, lithopone, zinc white, white lead in oil, rosin, linseed oil, turpentine, white spirit, wood oil .. .. .	114
Japan ..	Chemicals, dyes, paints, varnish, fertilisers .. .. .	67
United States ..	Benzolic acid and salts .. .. .	70

\* The Official Secretary, Commercial Information Bureau, Australia House, Strand, London, W.C. 2.  
† The Canadian Government Trade Commissioner, 73, Eastgall Street, London, E.C. 2.

**MARKETS SOUGHT.**—A Canadian firm wishes to dispose of 10,000 tons of fertilisers (ammonia and phosphoric acid compound).

A Canadian company invites applications for quantities of "Stellite" (high-speed steel).

[Inquiries to the Canadian Government Trade Commissioner.]

### TARIFF, CUSTOMS, EXCISE.

**Australia.**—The import of calcium carbide is prohibited except under licence as from Dec. 2, 1920.

The prohibition of the export of pig-iron and manufactures of metals has been revoked.

**Egypt.**—Among the articles that may be exported without export licence (under certain conditions) are coconuts, sweetened condensed milk, dyes, gold leaf, nuts (except arachis), starch, edible fats, and margarine.

**Fiji.**—The export of copra is now permitted without licence.

**France and Algeria.**—The "coefficients of increase" on certain articles have been increased. Articles affected include glucose, chicory, vinegar, aluminium powder, iron shavings, bronze powder, soap, yeast, syrup, certain kinds of leather, and many chemicals and drugs.

**Germany.**—Among the articles hitherto temporarily admitted duty free which must now pay the prescribed rates of customs duty as from January 1 are vegetable wax, beeswax, spermaceti, copper sulphate, zinc oxide, ammonium nitrate, lead nitrate, cascine, certain kinds of leather, dissolved rubber, and soft rubber paste.

**Italy.**—Certain building materials, including cement, may be imported duty free until June 30, 1922.

**Latvia.**—Among the articles that may be imported free of restrictions are medicaments, cast iron, steel, copper, white metals, naphtha, engine oil, fertilisers, tanning materials, sole leather, window glass, and industrial chemicals.

Articles the import of which is prohibited include, *inter alia*, beer, gold, silver, bronze, glass and porcelain (with some exceptions), perfumery, toilet soap, yeast, syrup, certain kinds of leather, and certain pharmaceutical preparations.

The import of all other goods is allowed under special licence.

**Netherlands.**—Export prohibitions have been removed from acetone, bone meal, Chinese wood oil, coal-tar dyes, gypsum, lime, limestone, dyeing woods and extracts, medicines, petroleum, pyrites, rosin, asphalt, shellac, train oil, wood tar, pitch.

Recent customs decisions affect solid fats, diphenylmethane, rubber yarns, bromoform, betanaphtholmethyl ether, beta-naphtholethyl ether.

**Norway.**—The prohibition of the export of salt has been withdrawn.

**Poland.**—Among the articles that may be imported without permits are cocoa, alcohol, albumin, fertilisers, glue, animal fats and oils, wax, skina, certain kinds of leather, cork, certain oil seeds, certain building materials and ceramic wares, tar, pitch, anthracene, benzene, phenol, rosin, asphalt, gums, rubber, many chemicals, ores, metals and metal wares, cellulose, and certain kinds of paper. [The list is very long and is given in full in the issue for January 20.]

**South Africa.**—Opium and preparations thereof may only be imported under certain specified conditions with the permission of the Secretary for Public Health.

**Trinidad.**—All goods entitled to preference under the British Preferential Tariff must be accompanied by a certificate of origin. [January 13.]

**Tunis.**—The import of hinder twine and sulphur is now permitted.

**Turkey.**—The export is prohibited of, *inter alia*, sugar, certain building materials, charcoal, quinine, neosalvarsan, aspirin, gold, silver, and nickel.

## TRADE NOTES.

## BRITISH.

**St. Helena in 1919.**—Agriculture generally has been much neglected in favour of flax-growing, which is making good progress. There was, however, a short supply of fibre, as small and immature leaves had been harvested to take advantage of the high prices. The output from the Government mill was 40½ t. of fibre and 27 t. of tow, and two private mills produced 410 t. of fibre and 115 t. of tow. Imports and exports were valued at £44,084 and £30,878, respectively, the latter including 603 t. of fibre and tow worth £27,108. The increased area devoted to flax has led to a shortage of foodstuffs, which has had serious consequences. (*Col. Rep.-Ann.*, No. 1039, Aug., 1920.)

**Turks and Caicos Islands in 1919.**—The trade of the Turks and Caicos Islands improved in 1919, the imports being worth £38,343 and the exports £33,554, as compared with £39,276 and £25,594, respectively, in 1918. Exports of salt, valued at £24,117, showed a decline owing to competition from accumulated stocks of salt in the Mediterranean, and lack of freight. Large stocks of salt—over 2 million hushels—were on hand at the end of the year. The cotton exported amounted to 15 bales of lint and 94 bags of seed, worth £580. The development of the production of cotton, said to be of high quality, is promising, as an estate of 8000 acres in the Middle Caicos has been leased to an American company for cotton-growing. (*Col. Rep.-Ann.*, No. 1042, Oct., 1920.)

## FOREIGN.

**Chemical Trade and Industry in Argentina.**—Several chemical factories which were started during the war have had to close down on account of inability to face foreign competition, and, generally speaking, the production of chemicals is small and inefficient.

Three firms manufacture sulphuric acid (53°–66° Bé), using sulphur from Italy (by preference), Chile, and the United States; chemically pure sulphuric and hydrochloric acids are produced, but only commercial nitric acid. In spite of the difficulty of transporting sulphuric acid over long distances through the tropics, local manufacturers do not find it easy to compete with foreign exporters. Acetic acid and vinegar are made in the country, but glacial acetic acid is imported. Two firms are producing, near Cordoba, acetate of lime, lead acetate, acetic acid and acetone by the distillation of wood of the algarrobo tree, but these products have to be marketed at prices higher than those imported owing to the high cost of working on a small scale. Practically no tartaric acid is made in the country, but the possibilities are well worth investigating. There is a demand for both tartaric and citric acids, preferably in the form of crystals. Imports of carboric, boric, carbonic, hydrofluoric, and tannic acids are small. Imports of alum have declined from an average of 6300 tons in 1913–16 to 490 tons, and are now derived mainly from the United States. Liquefied ammonia is in demand for refrigerating plants, about 540 t. being imported annually, of which 90 per cent. is supplied by the United States. The yearly demand for industrial arsenic is about 250 t., although in 1919 more than double this amount was imported, again mainly from the United States. The demand for calcium carbide has decreased from 9000 metric tons in 1913 to 3000 t. in 1919. Glucose is in great demand, the imports averaging 2100 t. per annum. There is a small but insufficient production of electrolytic bleaching powder, and the annual importation averages 680 t. The imports of other chemicals in 1918 were:—Aluminium sulphate, 1155 t.; alum, 490 t.; anhydrous ammonia, 540 t.;

arsenic compounds, 250 t.; boric acid, 55 t.; barium sulphate, 2024 metric tons; copper sulphate, 1740 t.; iron sulphate, 10 t.; magnesium sulphate, 676 t.; glycerin, 110 t.; paraffin wax, 1811 t.; and rosin, 14,100 t. Most of these chemicals are obtained from the United States, but the requirements in sheep and cattle dips, some 7500 t. per annum, are derived almost entirely from Great Britain; stearine, tallow and similar products are obtainable locally. There is a large demand for disinfectants, veterinary remedies, tar oil, carbolic acid, serum, and drugs in general.

The first paper-pulp factory established in the country was expected to start operating in September last. The mill, situated near Barranqueras on the Parana river, and under German-Argentine control, utilises a perennial hog grass known as "paja brava," and has a capacity of three tons of strawboard per day. The plant will in time be increased in order to furnish a larger output of a better quality pulp. There is a good field for the wood-pulp industry in northern Argentina, as there is an abundant supply of suitable raw materials and a ready market for the products.

An Argentine company is now producing on a commercial scale goods made from flax-straw waste. The process used is new, and it is claimed that the product is equal or superior in colour, elasticity, length of fibre, and resistance, to fibres retted by the old methods, which required many days' time, as compared with less than half-an-hour by the new method. The estimated quantity of linseed straw available in Argentina is 54 million tons annually, and except for the small amount (25 tons per day) used by the company, the whole of it is burned. (*U.S. Com. Rep.*, Nov. 2, 18, 19, 27, 29, Dec. 2, 1920.)

**Paraguay in 1919.**—The republic of Paraguay has a population of over a million, but the buying capacity for foreign goods is limited to not more than a fifth of this number. Cattle raising, meat packing, agriculture, the production of quebracho extract, and some lumbering constitute the chief industries. Cotton of good quality and sugar are also produced but are not exported. The exports in 1919, worth about £2,963,223, included:—Essential orange oil, 38 metric tons; quebracho extract, 32,876 t.; and 256,958 hides. Argentina and Spain took 56 and 10·8 per cent. of the exports respectively. The imports, valued at £3,167,194, included chemicals, drugs, mineral oils, and paints valued at £161,099, and china, earthenware, and glassware worth £25,002. Of the total imports, Argentina supplied 42·8, England 24·7, and the U.S.A. 12 per cent. (*U.S. Com. Rep., Suppl.*, No. 48a, 1920.)

**Foreign Company News.—France.**—The capital of the Société Chimique des Usines du Rhône is to be increased from 3,200,000 to 21,600,000 fr. by the issue of 184,000 new shares of 100 francs each; that of the Cie des Salcs de Luzeac from 3 to 5 million fr.; and that of L'Azote Français from 1 to 5 million francs. During the year ended June 30, 1920, the Stéarinerie et Savonnerie de Lyon made a net loss of 385,386 fr., owing to the unsettled state of the market; its capital is now 8,000,000 fr. The Caoutchouc de Sumatra Cie. produced 435 metric tons of rubber during the year ended June 30, 1920, and made a net profit of £10,436; the output of the plantations is to be reduced by 25 per cent. (*Ind. Prod. Chim.*, Dec. 31, 1920.)

**Sweden.**—The adverse effect of high production costs on the Swedish chemical industry (*cf. l.* 1920, 3822) is further illustrated by the collapse of the A.S. Norsk Kemikalia of Oesterdalen. The company proposed to produce charcoal, tar, terpentine, wood spirit, and pharmaceutical chemicals, but owing to the excessive cost of construction wages and raw material, it decided to go into liquidation while its share-capital was still intact. (*Schweiz. Chem.-Z.*, Nov. 9, 1920.)

## REVIEWS.

THE DETERMINATION OF HYDROGEN IONS. By W. MANSFIELD CLARK. Pp. 317. (Baltimore: Williams and Wilkins Company. 1920.) Price, including postage, U.S.A. \$5, other countries \$5.50 net.

A book on the determination of hydrogen ions in the English language should find a welcome among workers in widely dispersed branches of chemical inquiry and application. It was known to the older chemists that numerous chemical processes comprising particularly those of importance to the biological sciences were dependent upon the "degree" of acidity or alkalinity of the media in which they took place, and it was also known that this degree was not amenable to estimation by direct titration, but was conditioned by the strength of the acids and bases present. In all these cases the determination of hydrogen-ion concentration furnishes a quantitative basis of investigation. Starting from the groundwork of the ionic theory, the subject under review, after having been worked out mainly in Ostwald's laboratory, was very greatly developed, especially in its biological applications, in Sorensen's laboratory in Copenhagen, and is now being vigorously prosecuted in its many ramifications, particularly in America.

The author of the present work, who is engaged in the research laboratories of the Dairy Division of the U.S. Department of Agriculture, and has himself contributed notably to the advancement of the subject, has here presented a book which furnishes a complete guide both to the theoretical and the practical side of hydrogen-ion determination. The main contents of the book include:—(1) An introduction, comprising a full theoretical discussion of acid and base equilibria, the  $pH$  scale of acidity and alkalinity, buffer action, etc.; (2) a number of chapters dealing with the colorimetric determination of hydrogen-ion concentration, the theory and application of indicators, the preparation and use of buffer solutions, protein and salt errors, etc.; (3) several chapters dealing with the electrometric method of hydrogen-ion determination, the theory of the hydrogen electrode, potential differences at liquid junctions, the apparatus and technique required for electrometric measurements, the sources of error, the relation of hydrogen electrode potentials to reduction potentials etc.; (4) a chapter on supplementary methods; (5) a chapter covering an immense range of applications; and (6) a bibliography with classified index containing about 1100 references.

The book deserves the highest praise throughout for clearness, trustworthiness, comprehensiveness, and interesting style. One might perhaps have wished to see the applications treated in greater detail, but when the number of references is realised readers will perhaps be inclined to agree with the author that it may be better to leave the detailed treatment of applications to the several sciences to which they belong. The author divides applications into two classes: (a) Those in which the phenomenon under investigation or control is seriously affected by the  $pH$  of the solution and which require the most accurate methods available. For these he recommends the use of the electrometric method. Obviously the difficulties to be met and corrections to be made increase greatly with accuracy required, and it would have been interesting to hear to what degree differentiation of potential has been hitherto usefully applied. (b) For those applications in which so high a degree of accuracy is not required the author recommends the indicator methods. The reviewer is inclined to believe that when the millivoltmeter system of

measurement is employed (which in the present book, as in most American publications, is ascribed to Hildebrand, but which was fully described two years before Hildebrand by the reviewer, in conjunction with Wood and Law, in its application to tan-liquors), the electrometric method has many advantages over the indicator method in cases where the total curve of neutralisation by acids and alkalis is important, and in which an accuracy of about one centivolt is sufficient. Thus, in the examination of soils one titration of the soil suspension with an acid and one with an alkali by the electrometric method will show not only the hydron concentration of an extract of the given soil, but also the hydron concentration which such an extract would show after known quantities of acid or alkali have been added to or have been formed in the soil.

The reviewer feels confident that, in spite of the large amount of work already done, there still remains a widening field of application for the determination of hydron concentrations; and also that the present book will contribute materially to the popularisation and extension of this important branch of physico-chemical measurement and inquiry.

HENRY J. S. SAND.

THE PRINCIPLES OF THE PHASE THEORY. By DOUGLAS A. CLIBBENS. Pp. xx.+383. (London: Macmillan and Co., Ltd. 1920.) Price 25s. net.

There is in many quarters to-day an increasingly clear perception, based to some extent on war experience, of the definite bearing of physical chemistry on many technological problems, and the present volume may be regarded as a product of this view. The particular field with which Dr. Clibbens deals has been cultivated more especially by the Dutch school of physical chemists—Roozeboom, Schreinemakers, and others—and has received all too little attention in Britain. On this ground, as well as on its own merits, the appearance of an authoritative work dealing with the phase rule in the English language is to be heartily welcomed.

The principles of the phase theory make possible a rational survey of the influence of temperature and concentration on the equilibrium condition of a material system, and it is in the resulting unification of treatment that the value of the phase rule mainly consists. A remarkable feature of the development of this subject has been the graphical methods, such as the equilateral-triangle representation and the projections of the space diagrams, devised to facilitate the recording of the complex phenomena involved. In this connexion there is an interesting remark made by Mr. F. A. Freeth, who contributes an introduction and to whom the volume largely owes its inception, viz., that the graphical methods introduced for phase rule purposes have served to emphasise the vital principle that *every point* on a diagram—not merely those on the curves—has a definite meaning.

Within the limits which Dr. Clibbens has set himself the present volume furnishes a detailed and thorough treatment of the subject, compiled by one who is evidently at home in these matters. The argument is on non-mathematical lines, and the development can be followed by a beginner, but at the same time he must be prepared for much patient work if he aims at achieving a real mastery of the subject. The power to interpret a complex phase-rule diagram is not easily acquired, and, in view of the concentrated attention which even a few pages of the present volume demand, it is scarcely suitable for the undergraduate student as an introduction to the phase rule. For the advanced or research student the book can be

heartily recommended, and it is matter for congratulation that such a volume is now available for English readers.

The author purposely limits the scope of the work to the consideration of condensed systems, including only one liquid phase, and that the only phase of variable composition. His view, with which most physical chemists will agree, is that a true comprehension of the phase theory can best be attained by a thorough study of the condensed system. Further, it should be noted that the experimental illustrations of the theory which Dr. Clibbens discusses are taken solely from the field of salts and aqueous solutions, although naturally the theory developed is equally applicable to all condensed systems.

Somewhat less than half the book is devoted to the consideration of condensed binary and ternary systems, while the remainder is concerned mostly with quaternary and quinary systems. Naturally, the graphical representation plays an all-important rôle, and the volume is profusely illustrated with some two hundred diagrams. As regards the text, the reviewer has the feeling that there is occasionally too much repetition. On the other hand, mastery of the principles and methods of the phase rule can admittedly be achieved only by the most painstaking attention to details, and a little over-repetition is not a serious fault in this case.

JAMES C. PHILIP.

**THE CARBOHYDRATES AND ALCOHOL.** By SAMUEL RIDGAL and Associates. Pp. 219. *Industrial Chemistry Series.* (London: Ballière, Tindall and Cox. 1920.) Price 12s. 6d. net.

This volume is one of a series designed to give a comprehensive survey of the chemical industries. The series, of which nine or ten volumes have already been published, has been conceived on somewhat novel lines, one of the aims being to present to the advanced student the reality of the living industry without confusing his mind with details and figures, which may be obtained from more highly specialised text-books. In certain respects this aim is admirably achieved in the present volume, and the sections on cane and beet sugars, which occupy roughly 100 pages, or about half of the book, give a clear, succinct, and eminently readable account of the commercial production of sucrose. A short history of the development of sugar production from the earliest times, and an outline of the physiology of the sugar cane and of the agricultural methods adopted, add greatly to the value of these sections.

Of the rest of the volume the impression gained is that an attempt has been made to cover too much ground. The subject of patent stills is dealt with in 25 lines, and the use of alcohol as a fuel in 20 lines. Beer and brewing occupy 20 pages, and only nine are allotted to wine, including the recovery of tartar. Thirty pages are devoted to grain spirit, potable spirits, industrial alcohol and synthetic alcohol.

Within these limits the subject matter is well and clearly dealt with, and the authors have contrived to get a large amount of information into a small compass, but the value of the book would have been greatly enhanced by a fuller treatment of fermentation and distillation. The authors have no doubt been restricted by limitations of space, but it would perhaps have been more satisfactory if the fermentation industries had been dealt with in a separate volume. It might then have been possible to include a few more diagrams and illustrations of plant, which in the sections on fermentation are conspicuous by their absence.

The production of alcohol by the hydrolysis of wood and subsequent fermentation of the sugars formed is included in the section on synthetic alcohol. This arrangement seems to call for re-

vision, as the term "synthetic" can strictly be applied only to alcohol produced from acetylene or ethylene, or by methods other than fermentation.

The book contains so many excellent features and is written in such a clear and attractive style that it is to be hoped that in future editions the treatment of the subject matter will be more evenly balanced, even if this involves considerable expansion.

G. W. MONTER-WILLIAMS.

**FIRST REPORT ON COLLOID CHEMISTRY.**—By arrangement with the Department of Scientific and Industrial Research, the First Report of the British Association on Colloid Chemistry and its Industrial Applications is to be reprinted and issued for sale by H.M. Stationery Office. Copies (price 2s. 6d., or by post 2s. 7½d.) may be obtained through any bookseller, or from H.M. Stationery Office in London or its branches in Manchester, Cardiff, and Edinburgh. A small stock is also held for sale at Messrs. Hortons, Harrison Street, Johannesburg, South Africa, and by the Government Printer, Wellington, New Zealand. The Report is no longer to be obtained from the offices of the British Association.

## PUBLICATIONS RECEIVED.

**FACTORY ADMINISTRATION AND COST ACCOUNTS.** By E. T. ELSOURNE. *New edition.* Pp. 811. (Longmans, Green and Co., 1921.) Price 4s.

**THE TESTING OF DYE-STUFFS IN THE LABORATORY.** By C. M. WHITTAKER. Pp. 100. (London: Heywood and Co., Ltd. 1921.) Price 12s. 6d.

**THE FUNDAMENTAL PROCESSES OF DYE CHEMISTRY.** By H. E. FIERZ-DAVIN. Translated by F. A. MASON. Pp. 240. (London: J. and A. Churchill. 1921.) Price 21s.

**LES ÉTHERS CELLULOSIQUES. VOL. I.: LA NITRO-CELLULOSE ET LE CELLULOÏDE.** By A. DUBREUIL. Pp. 334. (Paris: A.-D. Cillard. 1920.) Price 45 fr.

**CHEMISTRY OF PULP AND PAPER MAKING.** By J. SUTERMEISTER. Pp. 479. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1920.) Price 33s.

**HANDBOOK OF METALLURGY. VOL. I. COPPER-LEAD-SILVER-GOLD.** By CARL SCHEER. Translated by HENRY LOUIS. Third edition revised. Pp. 1171. (London: Macmillan and Co., Ltd. 1921.) Price 40s.

**LUBRICATING AND ALLIED OILS.** By E. A. EVANS. Pp. 128. (London: Chapman and Hall, Ltd. 1921.) Price 9s. 6d.

**THE PRACTICE OF LUBRICATION.** By T. C. THOMSON. Pp. 607. (New York and London: McGraw-Hill Book Co., Inc. 1920.) Price 36s.

**THE NATIONAL PHYSICAL LABORATORY. COLLECTED RESEARCHES. VOL. XIV., 1920.** Pp. 380. (London: H.M. Stationery Office. 1920.) Price 25s.

**PUBLICATIONS OF THE UNITED STATES BUREAU OF MINES, Department of the Interior.** (Washington: Government Printing Office. 1920):—

**BAUXITE AND ALUMINIUM IN 1919.** By J. M. HILL. ARSENIO, BISMUTH, SELENIUM, AND TELLURIO IN 1919. By J. M. HILL.

**PLATINUM AND ALLIED METALS IN 1919.** By J. M. HILL.

**LITHIUM MINERALS IN 1919.** By H. INSBLEY. FUEL BRIQUETTING OPERATIONS IN THE LARGEST CITIES IN 1919. By J. MIDDLETON.

## THE DRAFT HOME OFFICE REGULATIONS FOR CHEMICAL WORKS.

(From the Association of British Chemical  
Manufacturers.)

The provision of hygienic conditions for labour and of protection from danger for workers is now recognised as being absolutely essential in every industry, and especially is this so in the chemical industry where the work involved in certain manufactures is of a dangerous and unhealthy character. The draft Regulations for Chemical Works recently issued by the Home Office have set a very high standard for the conditions of employment to be maintained in chemical factories. This is very desirable, but it is felt by a large section of the industry that some of the proposed demands are of an extravagant and impracticable nature. In the larger and more modern works the provisions made for the health and general welfare of the employees are in most cases excellent, but the enforcement of the proposed regulations will, even in these instances, involve additional expenditure. It is therefore to be regretted that the new regulations are being introduced at a time when the financial position of the industry cannot bear the additional expense which will be incurred in carrying them out. This is particularly true of the smaller chemical works engaged in branches of the industry which are at present in their infancy, such as fine chemicals, intermediates and dyes, and it is generally recognised that at the present time it is not in the interests of the industry at large to place upon those works additional burdens unless they are absolutely essential to the welfare of the worker.

Part I of the Regulations applies to all chemical works and includes provisions for mess and washing accommodation, ambulance and rescue equipment. These provisions would doubtless conduce to the well-being of the worker, but they are framed on too lavish a scale. The demands made for the provision and maintenance of suitable protective clothing (Clauses 11 and 12) are of an extravagant nature unless they are restricted in their application to those persons *continuously* engaged in work involving *continuous* exposure to wet or dust or to substances of a corrosive or poisonous character. With regard to the supply of adequate bath accommodation (Clause 15), the employer has received very little encouragement in this respect in the past owing to the small advantage taken by the worker of the bathing facilities placed at his disposal. It is therefore only natural that the smaller manufacturer should feel reluctant to expend money on the installation of baths when it is probable that little use will be made of them by the employee. As regards Clause 21, which provides for accommodation for employees "whose work done standing," it is generally agreed that such provision is impracticable and open to abuse. It is further considered that greater detail is required in the wording of the regulations in order to enable them to be carried out effectively. In the provision relating to the use of life-belts and rescue apparatus, for example, it is essential that the size of the hole should be specified, and it is considered that the provision of an efficient exhaust-draught in the slaking (Clause 3 (h)) should only hold good when the quantity of material used in the process exceeds a specified limit. Clause 2, which refers to provision of adequate measures to prevent men from obscuring platforms, passages, etc., is obviously impracticable, unless it applies only when the surroundings are *continuously* obscured by the evolution of steam.

In Part II of the Regulations the provisions apply particularly to those works in which caustic pots are used and to places where chlorate, bleaching powder, dyes, and intermediates are manufactured. In these cases the regulations relating to washing facilities are naturally more stringent and involve the supply of baths, nail brushes, soap, clean towels, the provision of which, although desirable, is so readily abused in practice. It is, of course, generally recognised that all these precautions are absolutely essential in the manufacture of such irritants as dinitrochlorobenzene and TNT, and it is doubtless the experience gained in this respect during the war which has led to the inclusion of these provisions in the new regulations. It is to be feared, however, that according to the schedule of processes included under Part II, it will be necessary to satisfy these provisions in many cases where they are not absolutely necessary. The provision for the repair and cleaning of gloves (Clause 31 (a)) is regarded as impracticable, and it is considered that the washing facilities provided for in Clause 33 should be supplied only for those workers who are *continuously* engaged in the various processes mentioned.

It is generally felt in the industry that had the collaboration of such a body as the Association of British Chemical Manufacturers been secured in the compilation of the regulations they would have been found more generally acceptable to all sections of the industry.

## THE BRITISH GAS MANTLE INDUSTRY.

Among the industries that are seeking State assistance through the projected Government Bill relating to "key" industries are the gas-mantle and subsidiary industries. On behalf of these a statement has been presented to the Board of Trade by the Incandescent Mantle Manufacturers' Association, the main points of which are summarised below:—

The Gas Mantle Industry bases its claim to be regarded as a "key" industry upon the following considerations:—I. Both in time of peace and in time of war incandescent gas lighting is necessary for the adequate illumination of docks, railways, factories, etc. II. It is estimated that 32 million tons more coal would be required yearly to produce the same illumination if flat-flame burners were used instead of gas mantles. III. The "stripping" of coal gas has been found necessary to ensure adequate supplies of benzol, toluol, and other hydrocarbons for the manufacture of dyes, drugs, and explosives; such "stripped" gas can only be used as an illuminant in conjunction with the gas mantle.

The value of the industry from the standpoints of efficient illumination and coal conservation was recognised by Lord Balfour of Burleigh's Committee on Commercial and Industrial Policy, and recent legislation authorising the sale of gas on a calorific, instead of an illuminating, basis has further emphasised the importance of the gas mantle. In Germany the essential nature of this industry, including the raw materials therefor, has been recognised by granting special allocations of coal at the lowest price and supplies of gas which are otherwise restricted.

Before the war the rare-earth chemicals and the gas-mantle industry were mainly under foreign domination. The total output of Indian monazite sand went to Germany, and British mantle-makers had to purchase thorium nitrate abroad at a price



fixed by the Thorium Convention, a combine of German and Austrian manufacturers which was itself controlled by the chief German maker of gas mantles. Further, foreign mantles were sold in this country at prices 2—35 per cent. below English factory costs, and 23—70 per cent. below the selling prices in the countries of origin. In 1913 foreign importations amounted to between 45 and 50 million finished mantles and over 11 million partly finished (about 70 per cent. of our entire consumption), and the latter were marketed in such a way as to evade the payment of British income tax by the English branch of the German combine, the Auer Gesellschaft. In the same year the English production was 22 million mantles, of which 10 million was made by a company associated with the German combine.

In consequence of this foreign domination the home industry became very precarious; nearly all the factories were running at a loss, wages were low, and employment uncertain and unsatisfactory. Hence at the outbreak of war there was insufficient plant to meet the country's requirements, supplies of thorium nitrate were cut off, and there was a shortage of clay rings and other materials required by the industry. To overcome these difficulties the Indian monazite deposits were placed under British control, and five home manufacturers took up the preparation of thorium nitrate, although until the factories were ready the nitrate had to be purchased abroad at arbitrary prices which showed large profits to foreign suppliers. These factories can now produce all the gas mantles the country requires; and during the war they supplied the cerium fluoride and cerium oxalate needed for searchlights and tracer bullets. Prior to the war all the mantle rings required by the home industry were either imported from Germany or supplied by a German-owned company with branch works in this country. These works were purchased by a group of the chief British mantle-makers and so extended that they are now able to meet the country's requirements, although not yet in a position to produce the full range of designs which were offered by the Germans. Ramie yarn, which was also derived from Germany before the war, can now be supplied by British spinning plants in sufficient quantity to meet all requirements.

Owing to the extensions made to buildings and plant (at war prices), and to the establishment of three or four new factories, the output capacity of the home industry is now over 130 million mantles per annum, which is sufficient to leave a considerable surplus for exportation. The following data show the position of the British industry in 1913 and on October 30, 1920:—

	1913.	1919-20.
Output of mantles .. ..	32,000,000	82,322,000
Capital employed .. ..	£440,670	£1,525,710*
Workers employed .. ..	1843	3783

\* Exclusive of £350,000 invested in the thorium nitrate and other subsidiary industries.

The present annual consumption of gas mantles is valued at £1,750,000; £400,000 is paid in direct wages, and at least an equal sum as indirect wages to convert the imported raw materials into the products used by the industry, labour and wages entering into the cost of the finished article to a degree unusual in other industries. The raw materials used by the gas-mantle manufacturer include thorium nitrate and other chemicals, cotton, ramie, artificial silk, asbestos yarns, clay rings, and cardboard boxes. At the present time there is grave stagnation in the above trades, which is mainly due to the increasing importation of foreign mantles (over 10 million in 1920), and the prospect of further competition at prices below British manufacturing costs. The low prices of foreign mantles are the result of:—(a) Attempts by foreign makers to recapture their former markets at any cost; (b)

the low rate of foreign exchanges; (c) longer working hours in foreign factories (60 hours or more per week compared with 48 in this country) and the lower standard of living of the worker; (d) large pre-war stocks of certain raw materials held by German manufacturers. The British consumer is not obtaining the full benefit of the lower prices, as the intermediate profit is mainly retained by the importers and dealers. The Labour representatives on the Joint Industrial Council for the industry proposed a resolution, which was passed unanimously, deploring the increasing importation of foreign mantles, and urging the Government to take steps to counteract the effects due to foreign rates of exchange. At the present time some of the British factories are closed down, and the rest are working short time; manufacturers are carrying large stocks at a season when demand normally exceeds the rate of production; employment in the industry is declining, and generally the situation is so serious that immediate action is imperative. In conclusion, it is submitted that the manufacture of thorium and rare-earth salts, incandescent mantles, and clay rings be recognised and assisted as "key" industries; that legislation be introduced forthwith to protect the British worker against unemployment resulting from foreign competition; and that the British manufacturers who have responded to appeals to lay the foundations of a self-supporting industry be assured a market free from unfair competition.

## THEORY AND PRACTICE IN CHEMICAL INDUSTRY,

With special reference to Physical Chemistry.\*

E. B. R. PRIDEAUX.

Although the principal difficulties under which chemical industry is labouring in this country are political and social, and therefore beyond the control of industrial organisers and chemists, there is a minor handicap, the neglect of theory, which can be overcome by the efforts of individual workers. The most successful practical men are, unconsciously, often great theorists, but in most cases they would increase their efficiency by studying the theories which bear upon their processes—if only to distinguish between practicable and impracticable inventions, between theories which lead nowhere and others which embody useful working hypotheses. In building up a healthy industry able to withstand severe foreign competition, theory and applications must be closely allied. For new points of view and working hypotheses, the industry is dependent, as it always has been, upon the work of private investigators, and especially upon the research work done in universities and technical colleges. In his presidential address to this Society in 1904, Sir W. Ramsay related how a scientific friend once pointed out to him that most of the changes which had been introduced in industries had originated in universities. Why? Because the investigator is unfettered. If a man sets out to improve an existing process he will very likely succeed, but he will not revolutionise a manufacture.

Not the least useful part of the expenditure of the Department of Scientific and Industrial Research has been that applied to maintenance grants in aid of student research workers in pure science. The business community would do well to follow the splendid example of private munificence which has been set in Nottingham and provide donations, to be used more especially for increase of staff and

\* Paper read before the Nottingham Section on Dec. 15, 1920.



equipment, in order to give the research students of Nottingham the best possible chances of distinguishing themselves, and of becoming versed in, and putting to the test, a wide range of theories. Both in applied and in pure science theories are the "imponderables" which enable us to control ponderable phenomena.

Few will doubt the efficacy and value of the "common stock" chemistry, in which the essential has been so thoroughly sifted from the unessential, and the ways made so plain that a good grasp sufficient for application can be obtained in a three years' course. Much of this was, however, in its inception "physical chemistry," and most strange to the orthodox chemists of the early 19th century. Now a new science, or rather many branches commonly summed up as "physical chemistry," have grown on the "common stock," and the student, sometimes also the teacher, stands bewildered. How much of all this is to be mastered, how much left untouched? It seems probable that in these branches the great advances of the future will lie, as they are applied one after another to the hitherto unsolved, or only empirically solved, problems. It is in these uncharted regions that gold is to be picked up, not necessarily for private profit, but for the advantage of the whole nation and humanity; and the more uncharted the country, the more necessary is the guidance of theories, even if these are only provisional in character and if each leads only a short part of the way.

Hence any real success in applied science is only gained after a liberal training on the theoretical side. The successful inventor or improver in chemical industry must be able to assimilate the results of research in pure science, and he must be quick to see their bearings and able to work out their applications. The trouble is that it is difficult or impossible to discern the bearing of a theory on technical processes until it has been read and understood. The original papers are perhaps written in difficult language, an interpreter is required, and he is usually a teacher who introduces it into a text-book. But long before this the first fruits may have been seized by some industrial chemists who have been adequately trained to master and apply theories.

The tendency in works' laboratories is to tabulate the results of pure science and to work out their applications, and the newly-formed Research Associations are in many cases working along these lines.

This is perhaps a partial answer to the question asked by many entrants into the chemical profession: Of what use is physical chemistry to me, or what opportunity shall I have of practising principles which will perhaps require an inordinate amount of time and energy? If the young man of good will and energy will enter these studies, he will surely find in them sufficient interest to carry him on, and eventually they will afford him a greater choice of posts in the profession. Although he may consider himself rushed during his university career, actually he has then more time and available energy than afterwards when he is in full work. Many of the most useful branches of physical chemistry can be applied quite effectively to a first approximation with the aid of sound elementary mathematics. Indeed, it is not so much the mathematics involved as the abstract ideas which present the difficulty. For example, those of osmotic pressure, concentration action, solubility product, electrolytic solution pressure, ions, free and total energy, the EMF of chemical reactions, velocity constants, and thermochemistry all require careful consideration from several points of view before they can be assimilated. Some can grasp them at once in the abstract form, but the usual road to, and test of, comprehension is through the working numerical examples. By whatever means, the

essential principles ought certainly to be acquired in student days as so many foundations upon which complete buildings may later be erected.

It will be found that thus an added power and a deeper insight into technical problems are obtained. Even when processes have reached a high degree of perfection and are run according to rule, so that apparently they hardly need the supervision of a professional chemist, it will be safer to employ one who has gained this depth of insight. However stereotyped a process may become, it has to be grasped by each new generation, and a study of the theories is the surest method. Even long-established processes occasionally break down, and few chemical reactions are as perfectly controlled as a steam engine.

The great generalisations of physical chemistry are required for the solution of most problems as soon as the reactions are carried out on a large scale. For example, the behaviour with regard to changes of pressure, temperature, and concentration of systems consisting of a gas, one or two liquids, one or more solids is described and put on a quantitative basis by the phase rule and other deductions from thermodynamics. There are some works chemists who consider the phase rule as important a tool as the "slide rule." These theories, with the aid of applied physics, dominate the operations of distillation, crystallisation, extraction and filtration, just as applied physics dominates those of heat exchange, flow of fluids, grinding and mixing; and the mere chemical side of physical chemistry dominates those operations in which chemical equilibrium, velocity of reaction, and electro-chemical processes are most important. To take one example, the laws relating to the crystallisation of mixed liquids are of the first importance in ferrous and non-ferrous metallurgy. A study of the conditions of formation of eutectics, of maximum freezing-point, compounds and solid solutions of various types, are equally important in organic chemistry. Thus, in the manufacture of margarine the freezing-point diagrams of the fats stearin, palm oil, etc. have been studied in order to obtain a mixture of from three to six fats which melts at about 25° C.

In the examination of technical products physico-chemical or physical tests have largely replaced chemical analysis. Thus, in ferrous metallurgy cooling curves, heat treatment, and microscopic analysis give the most important information. In other branches of industrial analysis it is often a question of determining the amounts of two or more known constituents of a mixture. Two physical constants, such as the density and initial boiling-point, may be sufficient to characterise the mixture. Numerous examples may be found of these methods in recent issues of the *Journal*. Sometimes a single constant such as the optical rotation is sufficient. The use of the refractometer, for example, is becoming very prominent. A single drop of liquid suffices to give a number which distinguishes oils from one another or which reveals presence of admixtures. A useful example of the combined use of refractive index and other constants in hutter analysis is to be found in the work of Mr. H. D. Richmond, who also informed the author that the refractometer is largely used in the jam industry to determine the degree of evaporation of the syrup.

To illustrate the manifold applications of physical chemistry it is not necessary to go beyond the record of this Section. Reference need only be made to the work of Sand, Wood and Law, who have adapted the hydrogen electrode to determine the acidity of dark-coloured tan liquors; to that of Procter, who has investigated the effect of acids and alkalis upon gelatin and leather material from the physico-chemical point of view; to that of Archhutt on the skin effect in lubricants; and to that of Wilkie on the conditions of reduction of

arsenic compounds at a lead kathode, which are doubtless capable of interpretation in terms of reduction potentials and velocities of successive reactions. The author believes that the industries of the East Midlands are waiting for control chemists able to interpret the wealth of research which has long been available. Can anyone suppose that the last word has been said in the exploitation of gypsum deposits? or that the chemical utilisation of our splendid coal deposits is carried on as largely as it might be in the district? or that great economies in labour and material, as well as improvements in the products, are not possible in our textile industries so soon as these are fully staffed with chemists?

One of the best examples of the value of working hypotheses is afforded by colloid chemistry. The chemical formulæ of soaps are well established, reversible chemical equilibria are present, and a regular gradation is found through acids and salts, such as acetates and propionates, which behave as regular electrolytes, up to oleates which exhibit truly colloidal phenomena. The properties of soap solutions cannot be interpreted in terms of the ions of sodium and of free acid radicle, more or less hydrolysed, giving free alkali and uncombined acid, which is more or less in the colloidal state. The question has now been attacked by the methods of conductivity, osmotic pressure, viscosity, surface tension, microscopy, ultramicroscopy, and ultrafiltration. The results as summarised in the Reports on Colloid Chemistry are of outstanding importance to all makers and users of soap. The work of the founders of colloid chemistry, as well as that done on soap by Harkins, Hillyer, Spring, Pickering, Donnan, McBain, and many others, has now resulted in descriptive theories which will enable anyone trained in the principles to take up technical problems with a fair chance of success. Immediate practical results are numerous, such as the recommendation to use sodium oleate in cold solutions on account of its solubility, and sodium stearate in hot solutions on account of the persistence of its colloidal nature. In order to account for the relatively high electrical conductivity, relatively low alkalinity (low hydrolysis), McBain has postulated the existence of "ionic micelles"—aggregates of fatty acid ions highly charged as compared with usual colloidal matter, and not visible by the ultramicroscope. These particles functioning as protective colloids account for two of the principal uses to which soaps are put. As a small quantity of gelatin or other colloid prevents the coagulation of red gold sols by salts, so the soap colloid prevents the coagulation of dirt particles, or rather assists their passage into the dispersed state in which they are easily washed away.

The production of stable aqueous emulsions by the addition of small quantities of soap is explained in a similar way, but is still more precisely described by Donnan and his co-workers as due to the lowering of the tension at an oil-water interface by the addition of small quantities of soap. Here the entirely theoretical work of Willard Gibbs pointed out the way which has led to such eminently practical results.

\* \* \*

In connexion with the above paper, Mr. H. Droop Richmond communicated the following:—Dairying is one of the oldest industries; it is not usually regarded as a chemical industry, nor as affording much scope for the application of physical chemistry, but when it is considered that the raw material—milk—is an emulsion of fat, in the liquid or solid state, in an amphoteric colloidal solution of the soluble salts of a complex, fairly strong insoluble acid, no further argument is required to show that physico-chemical problems of the highest order occur in dairying. As an old industry, it is apparent that very many of the problems have been

worked out by empirical methods, but they are far from having been elucidated completely. We do not know for certain the state of the fat globules in milk, though I think that my work, undertaken in conjunction with my brother, has shown that they are liquid above the melting-point of fat, and solidify very slowly on cooling; and I think that there is little doubt that they are not surrounded by any membrane other than a colloidal layer condensed by surface energy. We have comparatively little information as to the variation of this layer with changes of temperature, especially when the temperature is raised to such a degree that irreversible changes take place in the serum, yet this fact has importance in dairying. The laws of the rate of motion of these globules when submitted to the force of gravity, centrifugal force, and irregular impulses are only partially known, though of the highest importance in cream separation and butter churning. The motion of single globules is comparatively simple, but in milk the globules vary greatly in size and the practical importance of the laws of their motion becomes greater as their complexity increases, owing to close packing and also to variations of temperature; and until their laws are fully elucidated the scientific design of separators and churos is impossible.

The amphoteric nature of milk is due to the presence of salts of polybasic acids, the hydrions of which have widely differing concentrations; that is to say, hydriion concentration in milk does not vary with acidity. Cheese-making, which depends primarily on enzymic action, which in its turn is influenced by the hydriion concentration, has been controlled largely by acidity determinations in the main satisfactorily, but there is no doubt that a more scientific application of physical chemistry would result in an important improvement. In the manufacture of condensed milk, the hydriion concentration is increased and, unless the proportion of buffer salts is increased, it becomes too great to enable the milk to withstand the subsequent operation of sterilisation without the separation of insoluble acid salts; and this raises an interesting physico-chemical problem. The problem of escape of dissolved gases from a colloidal solution, attended by great foaming, has considerable importance. In the manufacture of casein and milk sugar there is on the one hand the problem of the separation and drying of a colloidal precipitate, and on the other the crystallisation of the sugar from a solution containing crystallisable salts.

## CULTIVATION OF THE AFRICAN OIL PALM.

With special reference to the East Indies.

An interesting article has appeared under the above title in the *Bulletin of the Imperial Institute* (1920, 18, No. 2, 209–252). Hitherto nearly all the palm oil and palm kernels of commerce has been obtained from wild trees in West Africa. The palm oil is prepared by the natives mainly by primitive methods which frequently result in the production of a low grade of oil, and the kernels are usually extracted by cracking the nuts by hand, thus entailing a great waste of labour. Owing to the great demand for fats of all kinds at the present time, much attention has recently been devoted to the cultivation of the palm in plantations and the preparation of the palm oil and kernels under modern factory conditions. The article under review has therefore been written with the primary object of supplying information which will be of service to planters or companies who may contem-

plate the establishment of oil-palm plantations. It deals fully with the distribution of the oil palm and the present position of its cultivation in various countries, describes the palm and its chief varieties, the climatic and soil conditions best suited to its growth, and the methods of cultivation and harvesting. The question of yields is discussed, and estimates are given of the probable financial returns obtainable from a plantation. The following points may be mentioned as of especial interest.

The importance of the oil-palm industry in West Africa may be gauged by the fact that the total annual exports now amount to about 130,000 tons of palm oil and 400,000 tons of palm kernels. The greatest output is in Nigeria, and in 1919 more than 100,000 tons of palm oil and 216,913 tons of kernels were exported from that country. Palm-kernel oil is not produced for export in any part of West Africa except Nigeria, whence 3356 tons was shipped in 1919.

Comparatively little attention has been given hitherto to the cultivation of the oil palm in plantations in West Africa, but plantations have been made in the Cameroons and at Gazi in the Belgian Congo, whilst in San Thomé the palm is commonly planted as shade for cocoa. The production from the wild trees could be greatly increased by thinning out the trees and clearing the undergrowth round them.

The cultivation of the palm as a regular plantation industry has only been carried on up to the present on the east coast of Sumatra, where in 1919

	Number of bunches.	Total weight of bunches. Tons.
5th to 10th year .. ..	768	4.51
11th to 30th year .. ..	640	9.44
31st to 50th year .. ..	192	3.77

the area devoted to this crop amounted to 12,800 acres, of which 6900 acres was in bearing. The palm has been planted successfully on a smaller scale in the Malay Peninsula, and several large areas have recently been taken up in the Federated Malay States for its cultivation on a commercial scale. In Sumatra the trees usually begin to fruit at the end of the fifth year, but in some cases they bear a crop in the fourth year.

Little is known with regard to the yield of fruits from the different varieties of oil palm, and therefore it is not easy to recommend any particular variety for cultivation. Assuming that the yield is satisfactory, however, it is desirable that a thin-shelled variety should be selected which gives a high yield of oil from the pulp and a fair yield of kernels. If the nuts are to be shelled by hand, the thickness of the shell is very important, but is of less consequence if nut-cracking machinery is employed.

The oil palms on the east coast of Sumatra are regarded as probably all derived from one variety, but it is not yet decided with which African variety this kind is identical. The advantage of cultivation is shown by the fact that in Sumatra, as also in Malaya, the tree has proved remarkably precocious, yielding large quantities of fruit at a much earlier age than in West Africa.

Detailed information on the yields given by trees of various ages in the Sumatra plantations has been given by Dr. A. A. L. Rutgers in *Mededeeling van der Algemeene Vereeniging van Rubberplanters ter Oostkust van Sumatra* (Algemeen Serie, No. 6, 1919, and No. 8, 1920), and the tabular statements therein have been reproduced in a somewhat modified form in the article under review. These tables give statistics of the gross yield of bunches and of fruits, the percentage of pulp, nuts and kernels in the fruits, and the percentage oil content of the pulp and kernels. From these particulars the following conclusions have been drawn.

The estimated normal annual yield of a plantation in lb. per tree is given below for trees of different ages:—

	Number of bunches.	Weight per bunch.	Total weight of bunches.	Total weight of fruits.
5th to 10th year ..	12	13.2	158	53
11th to 30th year ..	10	33	330	165
31st to 50th year ..	3	44	132	66

The oil palms now generally grown on the east coast of Sumatra give a high yield of pulp (62 per cent. of the weight of the fruit) and a low yield of kernels (8 per cent.). The fresh pulp of the fruits contains about 55 per cent. of oil and the kernels contain about 50 per cent. These yields are regarded as very satisfactory.

In establishing an oil-palm planting industry on modern lines—that is, with the use of machinery for preparing the palm oil and cracking the nuts—it is estimated that it would be necessary for profitable working to plant an area sufficient to produce 6000 tons of fruit per annum. From the fifth to the tenth year the average annual yield of fruits per tree is 53 lb., and from an acre bearing 64 trees (i.e., planted 26 ft. x 26 ft.) the total yield would thus be about 1½ tons; the actual area required to produce 6000 tons of fruit would therefore be about 4000 acres.

The theoretical yields of fruits and oil from an acre of oil palms planted with 64 trees are given as follows:—

	Total weight of fruits. Tons.	Total weight of pulp. Tons.	Total palm oil. Tons.	Total weight of kernels. Tons.	Total palm kernel oil. Tons.
5th to 10th year ..	1.51	0.906	0.498	0.121	0.090
11th to 30th year ..	4.72	2.826	1.554	0.377	0.189
31st to 50th year ..	1.89	1.134	0.624	0.151	0.075

In calculating the profits obtainable from an oil-palm plantation, it is necessary to take into consideration not the theoretical yields quoted above, but the practical yields. With modern machinery it should be possible to extract 80 per cent. of the oil contained in the pulp, and the whole of that in the kernels. The practical yield, therefore, may be estimated as follows:—

	Palm Oil.		Palm Kernels.	
	Per acre.	Per 5000 acres.	Per acre.	Per 5000 acres.
	Tons.	Tons.	Tons.	Tons.
5th to 10th year ..	0.398	1990	0.121	600
11th to 30th year ..	1.243	6220	0.377	1880
31st to 50th year ..	0.499	2500	0.151	750

On the basis of these results, together with considerations of the cost of machinery, land, labour, and upkeep, and the market value of the produce, it is shown that oil-palm plantations are likely to prove more profitable than coconut estates.

## SOCIETY OF CHEMICAL INDUSTRY.

### NEWS FROM THE SECTIONS.

#### GLASGOW.

At an informal meeting held on January 12, with Mr. J. H. Young in the chair, short papers were read on "Gas Warfare" by Prof. F. J. Wilson, and on "The Use of Magnesium Oxychloride Cements in Ships" by Dr. J. G. Robinson. The first paper described the methods of gas warfare used by the enemy, the nature of the substances employed and their physiological action. Dr. Robinson outlined the development of the manufacture of magnesium oxychloride cements, described their use for ornamentation and mural

decoration, and concluded with an account of the application of composition flooring as a substitute for wood decking on board ship. When used in conjunction with hard-wood sawdust, these cements make an ideal decking, as they are very resistant to heavy wear and tear, are fire- and vermin-proof, and are better insulators than wood. They can be laid more thinly and more quickly, and are cheaper than wooden decks. The danger of corrosion and the means of preventing it were described, and the difficulties were shown to be due mainly to the presence of excess of water, which vitally affects the expansion and also the tensile strength.

#### EDINBURGH AND EAST OF SCOTLAND.

An informal meeting was held in the Hall of the Pharmaceutical Society, Edinburgh, on January 18, Mr. J. Rutherford Hill presiding. Prof. G. Barger presented a communication on the Micro-Dumas Method of Determining Nitrogen, the apparatus being demonstrated by Mr. Harington. Mr. B. D. W. Luff showed a device to avoid overstepping the end-point of a titration, and Mr. M. T. Hay described a safety device for Carius tubes. Mr. W. T. H. Williamson gave a short account of the present position of the theory of soil acidity, and described some recent methods of testing acid soils.

#### NEWCASTLE.

At a meeting held in the Chemical Lecture Theatre of Armstrong College on January 26, a demonstration was given of the froth flotation process for the washing of coal. Mr. A. Bury, of Skinningrove, read the introductory paper and Mr. Bicknell carried out the subsequent demonstration.

Mr. Bury briefly reviewed the development of the coal-washing industry and pointed out that all methods previously used had depended upon the difference in specific gravity between the coal and the associated waste. Such methods failed in treating fine material, and the need had long been felt for a complementary or supplementary method capable of dealing with these refractory fines. A successful method had been devised by the Minerals Separation, Ltd., which was now being worked in Wales on a commercial scale.

A special laboratory has been established at Skinningrove with machinery and apparatus capable of dealing with samples from 400 gm. up to 1 cwt., which is at the disposal of all interested in the subject in the Cleveland, Durham and Northumberland districts. During a year's work at this laboratory many coals have been tested and the process has proved of the widest applicability. A list of excellent results obtained on raw coal, washery refuse, pit refuse, dump material, picking waste, etc., was given. Most coals consist of a mixture of pure coal of low inherent ash, bone coal containing high inherent ash, and shale containing varying amounts of bituminous matter. It is possible by the proper application of the flotation process to separate these constituents one from the other and thus obtain:—(a) A clean product of low ash content which gives an excellent coke, (b) a bony coal to be used for fuel purposes, and (c) a shale free from coal.

In making the demonstration it was shown that when water is agitated with small quantities of certain reagents, numerous very fine air bubbles are formed. When the coal is added to such a mixture the air bubbles attach themselves to the coal particles, and when the pulp is brought to rest the bubbles rise to the surface carrying the coal particles with them, forming a froth in which the solid particles are supported; the shale particles remain suspended in the water or settle at the

bottom of the containing vessel. Mr. Bicknell demonstrated the efficiency and simplicity of the process, obtaining both clean coal and clean shale residues.

#### LIVERPOOL.

On January 24, a meeting of this Section was held at Midland Adelphi Hotel, Liverpool. Dr. E. F. Armstrong presided, and Dr. Stephen Miall read a paper on "Diseases of Occupation."

Dr. Miall stated that, broadly speaking, constant exposure to any chemical compound was likely to be injurious to health, particularly when the compound gave rise to dust, fumes, or gas. The best general preventive was undoubtedly an abundant supply of fresh air, and the old-fashioned, dilapidated works had compensatory advantages over modern factories inasmuch as broken windows let in air and the worker was given a variety of occupations. Poisoning by gases from gas and internal combustion engines was increasing, but in nearly all cases was due to ignorance or carelessness on the part of the worker. After describing some measures which had led to the practical extinction of phosphorus poisoning, Dr. Miall referred to the toxicity of turpentine as the cause of the poisonous effects due to inhaling fresh paint, and then dealt with the characteristics of metallic poisoning, notably by lead and lead compounds. The diminution of cases in the white lead industry was very remarkable, and was the outcome of 20 or 25 years' study and the steps taken by the Home Office. The prohibition of the use of lead in paints would not adequately solve the problem; the real remedy was fresh air. Painters should rub down old paint with sandpaper moistened with mineral oil. Lead pigments should always be supplied ground in oil or other medium, and adequate ventilation would do the rest. It was difficult to induce such organisations as the International Labour Office to regard the subject of occupational diseases from the modern medical standpoint, so obsessed was the public mind with the bad conditions which prevailed some twenty-five years ago, but which had since been remedied. The International Labour Office may do much good, but it will accomplish nothing in the sphere of industrial hygiene without the co-operation of those primarily affected.

#### BRISTOL AND SOUTH WALES.

A meeting of this Section was held on February 3, at Bristol. Mr. E. Walls presided, and Mr. D. T. H. Beaton read a paper on "The Brewing Industry," in which he traced the development of the art of brewing from the earliest times to the present day. It was stated that the origination of the art was ascribed to Isis in 1960 B.C., and that Herodotus had mentioned the production of beer from barley in 450 B.C. The annual production of beer was now about 36 million gallons, and the industry contributed £120,000,000 directly to the Exchequer, or 15 per cent. of the total national revenue.

The chairman announced that the annual meeting of the Section would be held on March 17, and that he would give an address on "Chemical Industry in Bristol."

#### MANCHESTER.

The fifth meeting of the session was held at the Textile Institute, Manchester, on February 4. Mr. John Allan presiding. A paper on "The Utilisation of Subsidiary Alkaloids," by Prof. F. L. Pyman, was read, in the author's absence, by the Hon. Sec., Mr. L. Guy Radcliffe.

In the paper Prof. Pyman briefly outlined the general process for the isolation of alkaloids. Consideration was given to the stereochemical differences existing among certain alkaloids and special

reference was made to atropine and the *lævo* form of hyoscyamine, an explanation being given of the transformation of the latter into the former. Reference was also made to pilocarpine and its relationship to iso-pilocarpine, and details were given of the chemical methods used for the methylation of the hydroxyl group and of the amido group, as well as methods of hydration in the case of ergotinine.

A considerable portion of the paper was devoted to the esterification of eegonine obtained from the erythroxylon coca bean, and it was shown how the lesser valuable alkaloids could be easily and technically converted into the valuable cocaine. Reference was also made to the opium alkaloids, and especially to the utilisation of narcotine and papaverine. The optically active varieties of the styptic adrenaline, and the constituents of the *Hydrastis Canadensis*, better known as the Golden Seal, were described, and special emphasis was laid upon the value of the main substance hydrastine as compared with berberine, the latter being of little or no medicinal interest.

#### AMERICA.

The Perkin Medal for 1921 was presented to Dr. Willis R. Whitney at a meeting held at the Chemists' Club, New York, on January 14, Mr. Sumner R. Church, chairman of the Section, presiding. Dr. C. F. Chandler, senior past-president of the Society in America, in making the presentation, outlined Dr. Whitney's career, alluding more particularly to his work as Director of the Research Laboratory of the General Electric Company at Schenectady, N.Y., a post he has held since 1900.

In electric lighting the first radical improvement in the carbon incandescent filament was due to Dr. Whitney's personal work. The "metallised" filament, or "Gem" lamp, which he developed, gave 25 per cent. more light for the same wattage than the standard carbon-filament lamp. Millions of these new lamps were sold in a single year. A little later the laboratory solved the problem of mechanically working tungsten, and showed how to make the drawn wire which has given the tungsten lamp its universal application. The latest achievement of the laboratory in incandescent lighting is the gas-filled, or half-watt, lamp, which in its larger sizes has twice the efficiency of the vacuum lamp, and nearly equals the most efficient arcs. In arc lighting the magnetic electrode has been developed, and with it the most successful arc lamp of to-day. The laboratory has produced many new insulating materials and alloys, new processes, like "Calorising," for giving metals protective coatings; new articles of manufacture like "sheath wire"; new materials like "water japan" and "Genelite"; new electric furnace products like boron carbide, useful as a flux in casting copper, and titanium carbide for arc-lamp electrodes; new laboratory apparatus, such as the Arsen vacuum furnace, the tungsten tube furnace, the Langmuir condensation vacuum pump, the Coolidge X-ray tube, etc. Dr. Whitney was president of the American Chemical Society in 1910, and president of the American Electrochemical Society in 1911. He was awarded the Willard Gibbs Medal in 1916 and the Chandler Medal in 1920.

Dr. W. R. Whitney then acknowledged the honour which had been conferred upon him and gave an address on "The Biggest Things in Chemistry," which contained, among other things, the following dicta:—

In America patents are granted only to individuals, not to companies or laboratories, although the discoveries upon which they are based are often the result of corporate work. In this instance the award is to be regarded as belonging not to the recipient but to the research laboratory which he directs. Perkin's life contained all the

data needed to analyse scientific research, and illustrates the fact that the great advances have usually been made by men trained in their particular line of investigation and working just beyond the boundaries of the known. Perkin's apprenticeship under Hofmann so imbued him with the spirit of scientific research that after years of commercial success he returned to it and enjoyed it for the rest of his life. The essentials for success appear to be the teacher of heuristic instinct, the laboratory, and the school training. It is incorrect to say that Perkins, Faradays, and Pasteurs are born, not made, for the process is entirely standardised. American schools have been working on the wrong lines, and instead of turning out men well trained in research they have been consuming energy in bringing large numbers of men only part of the way.

No two chemists would agree at first as to what constitute the most important things in chemistry, but on reflection many would admit that the biggest things are summed up in the word "possibilities." Chemistry has grown and survived because all its precepts and principles can be re-tested at any time and anywhere. It is a young science, but its rate of advance is almost incredibly rapid, and one feels that no one has ever had such possibilities open to him as the present-day student of chemistry. The process Perkin followed is that which has led to most of our discoveries; it is the encouragement of natural inquisitiveness under the best conditions; no short-cut and easy process would have produced dyes from tar. One of the biggest things in chemistry to-day is to learn how to make the teaching of chemistry *productive*, and a study of the lives of such men as Wöhler and Pasteur should conduce to this end. They teach the value of high training combined with faith, a combination seldom found in the individual American.

In inorganic chemistry the number of available metals has been doubled within a few years, so raising to the *n*th power the number of possible alloys. Many of these metals are coming into use. The isolation of calcium is less simple than that of zinc, copper, iron, or tin, and its properties are different, but as usual it is the differing properties which determine the new uses. During the war it was used as a generator of hydrogen to maintain a very high pressure of this gas inside certain deep-sea sound-detecting devices, the sea water itself acting as the other reagent. It is also used as a continuously reacting purifier for argon in the "Tungar" rectifier, which consists of a bulb made of a special new glass, a spiral of tungsten wire, an electrode of artificial graphite, a little argon, and some metallic calcium.

In the sphere of organic chemistry, is it not possible that a field will be developed directly from carbon itself as great as that of dyes from coal tar? The gates thereto were apparently opened when calcium carbide was first made; and when we reflect that this substance made possible the manufacture of acetone, alcohol, etc., we realise the possibility of making, by the same road, useful food as certainly as medicaments.

In agriculture we are attracted by the thought that our troubles with nature are sometimes due to our personal limitations, not to the limitations of nature; and there appears to be a fertile field for research into ways and means of reducing the amount of human labour expended upon the growing of crops. Some of the biggest things in chemistry are coming from research in biochemistry and hygiene.

The remainder of the address was an elaboration of the thesis that the spirit of research and the quest for truth in the experimental investigation of material phenomena lie at the very roots of human progress, both material and ethical.

## LONDON.

At a meeting held on February 7, at Burlington House, with Mr. Julian L. Baker in the chair, a paper on "The Erosion of Bronze Propellers" was read by Dr. O. Silherrat. Recent investigations by a sub-committee of the Board of Inventions and Research have confirmed the author's previous conclusion that the deterioration of bronze propellers is due to mechanical causes. However, the Committee's conclusion that erosion is due almost exclusively to the collapse of vacuum bubbles ("water-hammer action") is not in agreement with the author's results, which show that after the surface of the metal has been damaged by this action further erosion is chiefly caused by frictional rub of the water.

Chemical analysis of the eroded portions of propellers shows that the composition of the eroded alloy is unaltered. Slight etching extending over considerable areas of the metal, which is sometimes observed when the propellers first come out of the water, is due to frictional rub and can be produced by high-pressure water jets impinging upon the surface of the metal. A determination was made of the daily loss of weight of a portion of a propeller exposed to the action of sea water, and it was inferred that over 5000 years would be required to produce by chemical action the excessive erosion frequently observed. The effects of electrical currents due to leakage and to mechanical stresses set up in the metal were found to be negligible. Microscopic examination of eroded propeller surfaces and sections showed deformation and breakage of the crystals composing the  $\alpha$  constituent of the alloy, whilst those of the  $\beta$  constituent were practically unaffected. Macroscopic examination showed that the majority of eroded propellers were either small ones running at high speed or large ones transmitting great power, and that the presence of dirt in the castings was without effect. Investigation of many eroded propellers enabled a classification to be made based upon the location and extent of the areas eroded, each type being traced to conditions producing currents of water containing vacuum bubbles impinging upon the affected areas. Since the  $\alpha$  constituent of propeller alloys was found to be much less resistant to erosion than the  $\beta$  constituent, the author investigated the production and properties of new alloys, but found that a copper-zinc brass possessing only  $\beta$  structure possessed undesirable mechanical properties. By the addition of small percentages of other metals an alloy with homogeneous  $\beta$  structure was obtained which had all the good mechanical properties of the older bronzes, and propellers made of it were found in practice to be very resistant to erosion.

## MEETINGS OF OTHER SOCIETIES.

## SOCIETY OF PUBLIC ANALYSTS.

At the annual meeting held on February 2, Mr. A. Smetham delivered the presidential address and officers were elected for the ensuing year.

Mr. J. L. Baker and Mr. H. F. E. Hnlton described an iodometric method of determining the diastatic power of malts in which use is made of the quantitative oxidation of maltose to malto-hionic acid by iodine in presence of sodium hydroxide. Results obtained on the same samples by this method and by that of Ling were given. The advantages claimed for this method are the greater accuracy with which the iodine titration may be carried out as compared with Fehling's solution, the elimination of an external indicator, and the possibility of using artificial light for the final titration.

In a paper describing experiments on the use of extract of red squill (*Scilla maritima*) as a rat poison, Mr. F. W. Smith showed that the most effective form was that prepared in water from an alcoholic extract of the fresh bulb of red squill, and that salicylic acid could be used as a preservative without fear of hydrolysing the glucoside.

A paper on the composition of Harrogate mineral waters, by Mr. W. Lawson, contained a summary of the analyses carried out in connexion with Prof. Smithell's survey of these waters, analyses dating back some 90 years being also given for comparison.

## THE ROYAL INSTITUTION.

The first Friday Evening Discourse of the new session was delivered on January 28 by Prof. Sir James Dewar on "Cloudland Studies." The first part of the lecture was devoted to experimental illustrations of natural cloud effects produced with the aid of liquid air or liquid oxygen. This was followed by a discussion of the early work on solar radiation by Sir J. Leslie, D. Forbes, and Pouillet, and of the determination of the "solar constant," that is, the amount of heat energy measured in gram-calories which would impinge vertically on 1 sq. cm. of the earth's surface provided there were no intervening gases to intercept the rays. With the aid of his pyrheliometer, Pouillet did very accurate work, his value for the constant, 1.76, being within 10 per cent. of the result of the latest determination (1.93) by C. G. Abbott and his staff in America. Leslie and Pouillet also determined the zenith or black-body temperature of the sky, using different methods, but these early investigators were ignorant of several disturbing factors, such as the variation of the wave-length of the emitted energy with temperature. The maximum wave-lengths of the black-body radiation emitted at the respective temperatures of boiling water, solid carbon dioxide, boiling oxygen, nitrogen, hydrogen, etc., were given in a diagram which showed that at the lowest temperature yet attained with liquid helium— $2^{\circ}$  A.—the wave-length of the radiation should be  $1.465 \mu$  or  $1.465 \text{ mm.}$  The original investigators were also ignorant of the transparency of gases and liquids to different radiations, and in this connexion the lecturer performed an experiment to show that liquid air is diathermanous to low-temperature radiations, and described how he had used his thermoscope, which is based upon this principle (cf. J., 1920, 35 N), to determine zenithal temperatures on the roof of the Royal Institution, the lowest value obtained being  $-42^{\circ}$  C., in June last.

Observations made in America have indicated that the solar "constant" varies within about 5 per cent., a conclusion which was strikingly confirmed last year by Guthnick at the Berlin Observatory, who found that the variations in the intensity of the light reflected from Saturn were within 1 per cent. of the variations of the solar constant observed in America.

Dealing with the composition of the upper atmosphere at the end of his lecture, Sir J. Dewar stated that as yet we had no proof of the hypothesis that it consists of hydrogen and helium (and perhaps methane and traces of water-vapour), but the recent work of R. H. Goddard, of Clark University, Worcester, Mass., "On a Method of Reaching Extreme Altitudes" promised to solve the problem. Goddard's method consists, briefly, in sending up rockets containing multiple charges of very powerful nitro-explosive which are fired successively from the same combustion chamber. By this means a velocity of 7000 ft. per sec. can be obtained, and it should be possible easily to reach altitudes up to 50 miles. Theoretically, 200 miles should be attainable with 10–12 lb. of explosive, and 500 lb. of explosive should project the rocket into space.



It should not be difficult to obtain samples of the upper atmosphere by attaching to the rocket a suitable air collector, a mechanism to buoy it up for a short time at its maximum height, and a parachute to make it descend slowly. The efficiency of the Goddard rocket is about 50 per cent.

## CORRESPONDENCE.

### ORIGIN OF THE SOCIETY OF CHEMICAL INDUSTRY.

SIR,—With reference to Mr. D. de L. Herman's letter in your last issue, I had always regarded Mr. G. F. Davis as primarily responsible for the formation of the Society. I recall that while I was an assistant in the Chemical Department at Owens College, about the year 1873, there were frequent visits from Mr. Davis to Dr. Roscoe, and these visits no doubt related to negotiations, the objective of which was the formation of the Society of Chemical Industry.

The passage given below from the "Life and Experiences of Sir Henry Roscoe" (Macmillan and Co., 1906) explains these visits, and shows that the formation of a national rather than a local Society was advocated by Sir Henry, and thus the movement was given that broader outlook which has resulted in the present day world-wide extension of the Society. Speaking of the earlier enthusiastic workers, I would suggest that the name of Mr. Walter Weldon be added to those mentioned by Mr. Herman:

"The Society of Chemical Industry was founded in 1880. It originated with a few men interested in the Lancashire chemical trade, notably Eustace Carey, Ludwig Mond, and Edmund Muspratt. It was originally proposed that the Society should be a local one, the object being to bring together purely scientific men and those practically engaged in chemical industry in the Lancashire district. I felt strongly the importance of such a proposal, but I thought that if the thing were worth doing at all it should be a national and not a local institution. I gradually obtained adherents to this view, and the Society was founded on these lines. The first list showed that we had obtained already 360 members; at the present moment the numbers are over 4000. The history of the Society during the quarter of a century which it has existed has been one of constant progress and success, and its influence is felt not only in this country and our Colonies (Canada and Australia), but also in America, where we have one Section in New York and another in Boston.

"I was elected the first president, and many distinguished chemists, both scientific and industrial, have succeeded me in the presidential chair. The work which the Council has done in publishing a journal under the able editorship of my old pupil, Mr. Watson Smith, has been beyond praise."

Of the existence of the Faraday Club in Liverpool or of its activities I was not aware. It has an interest inasmuch as it shows that the example of the Tyneside chemists had spread to the Lancashire district. For in 1868 there was founded the Newcastle Chemical Society, of which Mr. I. Lowthian Bell, afterwards Sir Lowthian Bell, was the first president, and among his successors were the late Sir Joseph Swan, Dr. Lunge, and others well known to chemical industry. This society was active until 1893, when it was dissolved and transformed into the Newcastle Section of the Society of Chemical Industry, forming, in fact, the third Section of this Society.—I am, Sir, etc.,

P. PHILLIPS BEDSON.

Newcastle, Feb. 5, 1921.

## PERSONALIA.

Sir Robert Robertson, Director of Explosives Branch, Research Department, Woolwich, has been appointed Government Chemist in succession to Sir J. J. Dobbie.

Dr. R. Anschütz is to retire from the chair of chemistry in the University of Bonn at the end of March. The chairs of pharmaceutical and organic chemistry in the Czech University at Prague have been filled by the appointment of Dr. Sterba-Böhm and Dr. Plzak respectively.

The first triennial award of the Kelvin Gold Medal has been made to Dr. W. C. Unwin for his pre-eminence in the branches of engineering with which Lord Kelvin's scientific work and researches were closely identified. The medal was established in 1914 as part of a memorial to the late Lord Kelvin and in association with the window placed in Westminster Abbey to his memory by British and American engineers.

The death is reported from Paris of Prof. E. E. Bourquelot, who served forty-four years in the Faculty of Pharmacy and twenty years as general secretary of the Society of Pharmacy, Paris.

## NEWS AND NOTES.

### FRANCE.

**Industrial Notes.—Metallurgy.**—The most important event of the past fortnight has been the dissolution of the *Comptoir Métallurgique de Longwy* with its subsidiary *Le Comptoir d'Exportation des Fontes de Meurthe et Moselle*. In well-informed circles this dissolution is held to be the outcome of the restoration to France of Lorraine. Prior to the war the "Comptoir" had absorbed practically all the French producers of cast iron; but owing to post-war changes a complete re-organisation became necessary, with Lorraine in the foreground. As co-operation could not be achieved, the "Comptoir" concluded that it could no longer maintain the leading position it formerly held, by virtue of which it suppressed intermediaries, secured the necessary *clientèle* for members of the syndicate, and distributed orders among the different works in such a way as to minimise distances between the producing centre and the client. All such co-ordination tended to diminish cost prices. It is anticipated that this syndicate will soon be resuscitated in some new form and on an enlarged basis.

**Coal.**—The present surplus of coal is due less to improved production than to the industrial slump; it is probably purely temporary, and when trade revives accumulated stocks will quickly disappear. It is rumoured that Germany will notify the Reparations Commission that her present supplies to the Allies are so crippling her economical and industrial position that she will not be able to supply the 2,200,000 tons stipulated for 1921, nor the arrears of 500,000 tons for 1920 due in January and February of this year. In order to find outlets for coal when control is removed, agencies are to be opened at Paris, Nancy, Strasbourg, Châlons, and Dijon for the coal raised in the Saar district. There will also be an agency in Switzerland.

**Chemical Industry.**—German competition is increasing and becoming more open not only in chemicals but also in plant and laboratory appliances; and the differences in price are so great that efforts to resist it are unavailing. Thus thermometers, balances, etc., are being offered at prices 33–50 per cent. below French prices and are being delivered within three weeks instead of the 12–18 months required by French makers.



Great surprise has been caused by the announcement that La Compagnie Nationale de l'Azote, which is an offshoot of La Compagnie Nationale des Matières Colorantes, of Les Établissements Kuhlmann, of La Société des Produits Azotés, etc., is going to use the "Badische" method of producing synthetic ammonia instead of the Claude process which is giving very satisfactory results at the Grande Paroisse Works at Montereau.

A credit of 150,000 francs has been voted by the Chamber for producing resin under Government control in the State forests of south-western France, in the vicinity of Bordeaux.

The production of the Alsatian potash mines in 1920 shows a notable increase. The quantity sold amounted to 200,000 tons of pure potash ( $K_2O$ ), which represents one-fifth of the world's production in 1913.

Glass-making firms in France and in Belgium are very strongly affected by the present bad conditions in trade and by German and Italian competition. Several works have had to dismiss employees.

**Manufacture of Paper from Alfa Pulp.**—So far the United Kingdom has been the only country to use alfa, a product of French North Africa, for the manufacture of paper, but now, as a result of successful experiments, a French company is to be formed to produce the pulp. It is intended to acquire part of an explosives factory, situated near Avignon on the Rhone, and after 18 months to treat some 30,000 tons of pulp annually. The raw materials will be entirely of French origin, and the pulp produced will be dry, thus effecting economy in transport. Cellulose made from alfa is said to be suitable for the manufacture of smokeless powder. The exports of alfa from Algeria in 1917 were valued at 7,816,342 francs, of which the United Kingdom took 92 per cent., the share of France being less than 1 per cent. (*cf. J.*, 1918, 301 R, 476 R).—(*Bd. of Trade J.*, Jan. 13, 1921.)

#### AUSTRALIA.

##### Western Australia.

**Porcelain Works.**—Arrangements have been completed for initiating the manufacture of porcelain in Australia. In April next the Calyx Porcelain Co., Perth, will start erecting works at Subiaco which will cover three acres of land. Machinery to the value of £5000 has been purchased, and fifteen expert workers have been engaged.—(*Official.*)

**Petroleum Exploration.**—According to papers laid before the Parliament of Western Australia, the Anglo-Persian Oil Co., Ltd., has offered to form a Western Australian company with a capital of £100,000, of which £50,000 would be available for geological survey and boring. The company asked for legislation to be introduced next session to enable it to acquire leases of not less than 100,000 acres in the aggregate. The State Government acceded to the request for immediate legislation but would not consider leases larger than 600 acres each, to which the company replied that it was willing to explore for oil in Western Australia, but required large concessions similar to those granted to it in other parts.—(*Official.*)

##### Queensland.

**Wolfram and Molybdenite Mining.**—In order to save the wolfram and molybdenite mines near Cairns, the Chamber of Commerce of Cairns is urging the Commonwealth Government to acquire the properties as a national asset. During the war the deposits were exploited and the concentrates were shipped to the United Kingdom.—(*Official.*)

**The Sugar Industry.**—The twentieth annual report of the Bureau of Sugar Experiment Stations, issued December, 1920, states that never in the history of sugar growing in Queensland has there been so

large an area under cane. The spring has opened up most favourably, and the prospects for next year are highly promising. Last year the yield of sugar from the cane attained a record, and this year it is also good, viz., about one ton of sugar from eight tons of cane, equivalent to a total yield of 162,000 tons.—(*Official.*)

##### Tasmania.

**Electrolytic Zinc.**—The Electrolytic Zinc Co. of Australasia is making large additions to its plant at Hobart, where 1200 men are now employed. It is anticipated that the company will shortly be able to export annually 10,000 tons of zinc oxide, 15,000 t. of zinc-lead oxide, 10,000—15,000 t. of lithopone, large quantities of rolled zinc sheets, and metallic silver and lead. The present output of zinc is 25 tons per day, and by November next this should be increased to 50—60 t. The current consumption is now 5700 h.-p., but when the full 30,000 h.-p. contracted for is available, the plant will be able to treat 100,000 t. of zinc concentrates per annum. Extensive warehouse and wharfage facilities are being supplied in order to cope with the expected increase in the export trade.—(*Ind. Aust. and Min. Stand.*, Nov. 25, 1920.)

According to the *Times Trade Supplement* of Feb. 5, the above-named company has decided to cease production pending the increase of the plant capacity to 50 tons. The heavy fall in the price of spelter has led to this decision.

##### CANADA.

**Projected New Paper Mill in British Columbia.**—Plans are now practically complete for the erection of a large modern pulp and paper mill, to employ 1000 men, at Prince George, B.C., at a cost of \$6,000,000. The capital is being supplied by interests in Eastern Canada and the United States.—(*Official.*)

**Electric-Steel Smelting in British Columbia.**—A new company, the British Columbia Steel Works, Ltd., has been formed to amalgamate the electric steel plants at Port Moody, Eburne, and Tudhope. For the present the three will continue operating, but later the work will be concentrated at Port Moody.—(*Official.*)

"Canadian Chemistry and Metallurgy" is the new title of *The Canadian Chemical Journal*, which has been chosen mainly because the older name too closely resembled that of various foreign journals that cover somewhat different fields—for example, specialised research. It is also announced that an additional paper, to be called *Canadian Dyer and Color User*, will shortly be issued from the same office.

##### SOUTH AFRICA.

**Industrial News Items.**—Asbestos deposits in the Haenertsburg District, Transvaal, are to be worked by the South African Asbestos Mines, Ltd., a newly formed company with a capital of £50,000.

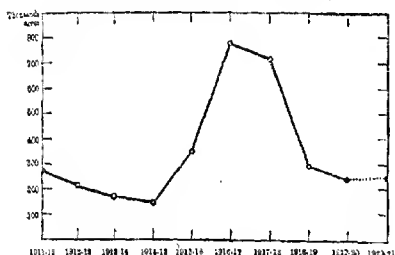
The fifth annual exhibition of the Chemical Metallurgical, and Mining Society of South Africa to be held at Johannesburg from March 9 to 21 will illustrate the latest developments in apparatus and machinery connected with chemistry, metallurgy and mining, and a section will be devoted to crude and manufactured products of the country.

The production of compressed oxygen and hydrogen will shortly be undertaken at Newtown, Johannesburg, by the Hydrogen, Oxygen and Plant Co., which will also make ammonia coils, condensers, etc., for refrigerating plants and undertake the welding of metals. A large works with two gasometers having a storage capacity of about 8000 cu. ft. is being erected at a cost of £35,000, and the plant will include 50 large cells capable of producing 3 million cu. ft. of gases yearly.—(*S. Afr. J. Ind.*, Dec., 1920.)

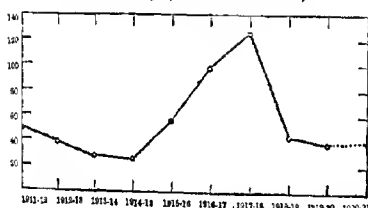
## BRITISH INDIA.

**The Indigo Industry.**—The final general memorandum on the indigo crop for 1920-21, given in the *Indian Trade Journal* of December 31, 1920, states that the estimated total area under indigo is 238,400 acres, and the total yield 40,400 cwt., representing increases of 1·3 and 7 per cent., respectively, over the finally revised estimates of last year. The season was unfavourable, except in Madras, and the condition of the crop was reported to be fair. Of the total area under indigo in British India, 54·3 per cent. is in Madras (106,800 acres), 20 per cent. in the United Provinces, 14·7 per cent. in Bihar and Orissa, 9·3 per cent. in the Punjab, 1 per cent. in Bengal, and 0·6 per cent. in Bombay and Sind. The accompanying charts show the variations in the present figures of area and yield as compared with the finally revised figures of the last nine years.

Area under Indigo (in thousands of acres).



Yield of Indigo (in thousands of cwt.).



The exports by sea to foreign countries in each of the last five years have been (in cwt.):—1915-16, 41,932; 1916-17, 34,230; 1917-18, 31,062; 1918-19, 32,707; 1919-20, 32,687.

The imports of synthetic and natural indigo into the United Kingdom during the last five calendar years have been:—

	Synthetic indigo.	Indigo.	Total.
1915 ..	11,240 ..	25,157 ..	36,397
1916 ..	4618 ..	30,527 ..	35,145
1917 ..	11,966 ..	13,501 ..	25,467
1918 ..	2084 ..	5287 ..	7371
1919 ..	2466 ..	3701 ..	6167
1920 ..	4541 ..	1603 ..	6144

Of the total amount of natural indigo imported into the United Kingdom in 1919 (3701 cwt.), 2357 cwt. came from India, 445 cwt. from San Salvador, and the rest from other countries.

## UNITED STATES.

**A Museum of Chemical Types.**—It is announced that the Smithsonian Institute has accepted the request of \$25,000 left by Morris Leeb, chemist, for the establishment of a "Loeb collection of chemical types," which will include specimens of all new chemical substances and make them available to research workers for reference purposes. An advisory committee of chemists will be formed to deal with requests for the use of type material from the collection.—(*Oil, Paint and Drug. Rep.*, Jan. 3, 1921.)

**Potash in 1919.**—The situation in the potash industry was very serious at the beginning of 1919. The high prices ruling during the war period had resulted in an increase of the annual output capacity to 100,000 short tons of pure potash (K<sub>2</sub>O), and the invested capital was estimated at \$25,000,000. Few of the larger plants had been paid for, and many were still under construction or had only been operated for a short time. One-third of the 1918 production was still unsold, prices had dropped from \$5 per unit in November, 1918, to about \$2·50, and the market was dull. Hence most of the producers closed down, at least temporarily, early in 1919, and some went out of business. Efforts were made to induce Congress to pass protective legislation but without success. As, however, imports remained small, prices were maintained and domestic output was readily absorbed.

Production in 1919 amounted to 110,243 short tons (28% K<sub>2</sub>O) from 77 plants, compared with 207,636 t. from 123 plants in 1918. The alkali lakes of western Nebraska again provided the largest supply, but the output from the alunite deposits in Utah diminished, and the extraction from silicate rocks in Wyoming was suspended owing to technical difficulties. Only one kelp plant in addition to the experimental plant of the Bureau of Soils at Summerland, Calif., reported production. The Government has continued its experimental work, and the Bureau has made a survey of the occurrence of borax in potash materials. Few developments occurred in the production of potash from molasses-distillery waste, beet-sugar refineries, or wood ashes, and no production from wool washings was reported. The total production in 1919 was constituted as follows:—Nebraska lakes 29·2%, other brines 36·6%, Steffens waste water 11·7%, molasses-distillery waste 9·1%, alunite 7·4%, cement works 3·8%, wood ashes 1·2%, blast-furnaces, kelp, and silicate rocks 1·0%. Exports were very small, and imports amounted to 39,619 short tons (K<sub>2</sub>O), compared with 7957 t. in 1918, and 270,720 t. in 1913.—(*U.S. Geol. Surv.*, Dec. 8, 1920.)

**Maize Oil.**—Bulletin No. 904 of the U.S. Department of Agriculture contains an account by A. F. Sievers of the production and utilisation of corn (maize) oil in the United States, where it has been manufactured on a commercial scale for about 25 years. The production in 1918 was 49,545 long tons, of which about 70 per cent. was refined for edible purposes.

The oil is derived from the germs, which represent about 10 per cent. of the weight of the grain and contain about 50 per cent. of oil; the inclusion of the oily germ in the various products of maize-milling renders the latter liable to become rancid, so that removal of the germ is desirable; this is carried out on a large scale, though whole grain is still milled, particularly in the Southern States. Degermination is effected by dry-milling in the manufacture of hominy products (grits, flakes, meal, and flour) and by the wet process in the manufacture of starch and glucose.

In the dry process the cleaned grain is sprayed with water and treated with steam till it contains about 20 per cent. of water, and is then passed through a degerminating machine, consisting essentially of a drum tapering horizontally, studded with cone-shaped projections, and revolving at 700 revs. per minute inside a casing with similar projections; in passing through the machine the kernel is torn and shredded, the husk and germ are loosened, but the starchy portions of the grain are not ground appreciably. After separation of the various products the germs are dried until the moisture content is about 14 per cent. In the wet process of degermination the grain is steeped for 36–40 hours in a 0·2 per cent. solution of sulphurous acid in wooden vats, which are in some cases arranged

so that the steeped grain can be removed from the bottom of the vat and fresh grain charged in continuously. The steeped grain is then drained and passed through mills which shred it, loosen the husks from the endosperm, and free the germs. The latest type of wet-degerminator consists of two circular vertical plates mounted (inside a casing) on a horizontal axis. Only one plate is geared to motor and revolved; in earlier types both plates were revolved.

The shredded grain is mixed with a large bulk of water and run to floating-vats, where the germs rise to the surface and pass over the end of the vats together with starch, which is removed by washing in perforated reels; the wet germs are then pressed to remove excess moisture, and finally dried to about 15 per cent. of moisture in steam-heated rotary driers.

Germs produced by the dry process are mixed with a good deal of the other portions of the grain; they contain an average of about 18 per cent. of oil and yield a cake with 5-7 per cent. of oil; wet-process germs, being more completely separated, contain about 45 per cent. of oil and yield a cake with 8-12 per cent. of oil.

The processes of oil-crushing and refining are similar to those used for other oil-bearing materials, though it is of interest to note that hydraulic presses have been almost entirely replaced by expellers which are more economical to run, partly owing to the present high cost of press-cloths; solvent extraction is sometimes employed. The oil cake is generally mixed with other products of maize-milling and sold for feeding purposes.

Crude maize oil is usually of good quality and low acidity, oil from dry-process germs being in some cases suitable for edible purposes without refining, and the average free fatty acid content of oil from seven mills being 1.11 per cent. by the dry process and 2.39 per cent. by the wet process; the acidity may, however, rise much above these figures, and oil from the 1917 crop averaged nearly 10 per cent. owing to the grain having been immature.

Maize oil has been used for many purposes, one of the chief being in the manufacture of rubber substitutes; it is still employed in soap-making, but most of the oil now produced is refined for edible purposes, the demand for which is evidently increasing. The elimination of the brewing industry, which is a large user of maize products, may cause a decrease in the production of the oil, though the increased demand for "corn-sirup" and other sugars may lead to increased milling of maize.

**Bauxite and Aluminium in 1919.**—The quantity of bauxite marketed in the United States in 1919 was 376,566 long tons, valued at \$2,201,747 at the mines, a decrease of 38 per cent. in quantity and 36 per cent. in value compared with 1918. Arkansas, with an output of 333,490 t., was the chief producing State. The domestic bauxite marketed was utilised as follows:—For aluminium 272,270 t., for chemicals 67,842 t., for abrasives 35,395 t., and for refractories 1059 t. Imports of bauxite amounted to 6082 t., and exports to 17,701 t. Aluminium production, valued at \$38,553,000, showed a decrease of 6 per cent. compared with 1918, which was due to accumulation of stocks. It is reported that plants for producing both aluminium metal and manufactured articles have been extended. The Aluminium Co. of America is building new plate mills at Alcoa, near Maryville, Tenn., and is enlarging the mills at New Kensington, Pa. Plants for making aluminium foil and powder have also been enlarged. There are now 23 plants producing aluminium sulphate, of which five are at municipal works. The total output of aluminium sulphate in 1919 was 200,034 short tons, and the average price was \$33.80 per ton; three plants pro-

duced 4806 short tons of the chloride and 8 plants produced 17,091 t. of alums, mainly ammonium and sodium alums (*cf. J., 1920, 288 n.*)—(*U.S. Geol. Surv., Aug. 30, 1920.*)

#### GENERAL.

**The Association of Swiss Leather Chemists.**—The Association Suisse des Chimistes de l'Industrie du Cuir was founded in 1918 as an independent association with objects similar to those of the Society of Leather Trades' Chemists. It has now about fifty Swiss members. At the last annual meeting, held in Lucerne in 1920, the Association decided, among other things, to take up the fight against the warble fly pest in Switzerland, and at a special meeting, held on January 15 last, the president submitted a scheme for the appointment of a Swiss committee to investigate this problem as part of the larger question of hide and skin deterioration. The scheme was accepted and a committee appointed. The Swiss Government and the Swiss Farmers' Association were represented at the meeting, and the president intimated that he had received a letter full of encouragement from the secretary of the Warble Fly Committee of the British Ministry of Agriculture and Fisheries. Another question discussed was that of official analyses in the leather trades, with a view to forming a special committee to study this subject in Switzerland, if necessary in collaboration with the existing committees of the International Associations.

**The Dye Industry in Spain.**—The Spanish coal-tar dyo industry, centred in Catalonia, developed considerably during the war owing to the absence of German competition. Two old-established firms in Barcelona—J. Pellicer and L. Sagnier—are now producing Delta, Congo, and other standard colours on a competitive basis, but neither these firms nor any of those recently established can produce the special dyes for which Germany is famous. The most important new companies are the Sociedad Española de Productos Químicos (*cf. J., 1919, 32 a*), which is supposed to control the market in Sulphur Blacks, and the Industria Química Nacional, which so far has only manufactured sulphuric and hydrochloric acids, etc., but is negotiating with German firms with a view to acting as their agents on the Spanish and South American markets. The Anilinas y Productos Químicos company, of Barcelona, is reported to be closing its works, as it is unable to produce at a profit owing to lack of raw materials and personnel; and the Química Tarasense company mainly produces chrome colours from raw materials imported from America. Except in the case of the simple coal-tar colours, it may be said that the recent efforts of the Spanish industry have come to grief, and that German colours still retain their supremacy.—(*Z. anorg. Chem., Nov. 26, 1920.*)

**The Glass Industry in Lorraine.**—The glassworks of Lorraine are situated at Dreibrunnen (near Saarburg), Chateau Salins, Forbach, St. Aurin (near Lörchingen), Götzenbruck, Meisenthal, and Münzthal (near Bitsch). The three last are the most important. The Münzthal works is now producing ordinary glassware, fancy wares being in small demand; it has the largest glass furnace in the world, with 24 pots. The Meisenthal factory, employing 500 men, has been doing well, and has recently spent 500,000 fr. on plant extensions, etc. The Götzenbruck factory is the largest spectacle-glass works in the world; it employs 800 men, but some of these have been dismissed owing to trade depression; it also manufactures watch and window glass. The Lorraine glass industry, although prosperous on the whole, has been affected by its isolation from the German market, and part of its trade has been lost to Germany and Japan.—(*Z. anorg. Chem., Dec. 24, 1920.*)

**German Chemical Industry in 1920.**—During the first half of the year the production of chemicals was very seriously affected by shortage of raw materials and coal, and the foreign demand, stimulated by the low rate of exchange, could not be satisfactorily met. These conditions created a danger of German chemical firms coming under foreign domination. The large companies met the altered circumstances by making large increases in capital and at the same time drawing themselves into closer association. Owing to this increase, the total capital of the most important German chemical companies amounted to 1.9 milliards of marks at the end of 1920. The very sudden upward move in prices of chemical products in January-March gave rise to the most rampant speculation, but ended disastrously for both home and foreign speculators after the Kapp insurrection. It would have been much better if the development of the export trade had taken place more gradually, as the sudden violent demand could not be satisfied owing to want of coal. In the latter part of the year, when coal supplies began to improve, production could not be developed owing to difficulties in selling. Many foreign contracts were cancelled as the value of the mark rose, and the threat of a trade crisis, stimulated by the reported sudden drop in the prices of foreign chemicals, has persisted over the end of the year. In fact, the present year opens with prospects of a crisis more serious than any which has been experienced since the 1860's. There is a doubt whether recent dividends were justified, paper money has never been so depreciated, and never has the necessity for ample reserves to cover repairs and replacements been so vital. The dividends of 14 to 20 per cent which were declared in spite of the large and growing demands for State taxes and workers' wages, were designed to support the heavy increases in capital; they were only justifiable as a division of war profits, after writing off the cost of extensions and new plant.

Problems connected with the re-organisation of the chemical industries are being dealt with by the Reichsarbeitsgemeinschaft Chemie, Berlin; the most important of these was that of coal supplies, for owing to the deliveries due to Allied countries under the peace treaty the requirements of German industry could not be met by more than 50 per cent at most. The coal-tar colour and soda industries, which employ, either directly or indirectly, over 3 million workers, were very hard hit. The Solvay works at Wurselen and Rheinsberg were shut down in January for want of coal, and similar trouble extended through all branches of the industry during the whole spring, and only in the summer did any improvement set in. Closely dependent on the coal problem is the supply of raw materials such as coal-tar products, salt, lime, alcohol, etc. These created grave difficulties in the past year, and the prospects for 1921 are not much better, since large quantities of benzol, coal tar and ammonium sulphate have to be delivered to France under the agreement of January 10, 1920.

The question of the socialisation of the coal mining industry is a disturbing factor, and a resolution condemning the proposal was passed unanimously at a meeting of the chemical trades held at Munich in September. The tendency of the proposal is to reduce production in the mining industry and to increase prices, thus crippling the chemical industries in their endeavour to rehabilitate German trade and meet foreign competition in the world's markets. The compulsory control of the chemical industry, which so rigidly enforced during the war, has now been abolished for internal trade, but it was reported on November 24 that restrictions on the export trade should be continued for a time. In any cases export licences have already been abolished.

Wages and salaries were increased two- or three-fold over those paid in 1919, but recently difficulties and disputes have arisen in the attempt to reduce wages by 10—15 per cent. There is little sign of the hoped-for fall in the cost of living, and demands for further increases in wages must be expected.

Organisation in chemical industry has developed to a remarkable extent during the past year, and industrial scientific research has been furthered by the foundation of many new institutes and societies. The exclusion of Germany from many of the world markets makes it imperative that the development of her chemical industry should proceed on the lines of concerted action and high-quality production.—(*Chem.-Zeit.*, Jan. 1, 1921.)

**The Beet-Sugar Industry in Bulgaria.**—As yet only beet sugar is produced in Bulgaria, but experiments with sugar cane are now in progress, and prospects appear to be favourable. Although the area planted to sugar beets in 1919 was only 21,414 acres, a decrease of 13,167 acres on the 1918 area, the total production was 11,861 metric tons, an increase of 8465 t. There are refineries in Sofia, Rustchuk, and Gorna Orchovitza, each with a capacity of 120 to 150 metric tons of raw sugar per 24 hours. No sugar is exported, but 4953 t. was imported in 1919, mainly from Czechoslovakia.—(*U.S. Com. Rep.*, Dec. 6, 1920.)

**The Mineral Output of Italy in 1919.**—The Italian mining industry made considerable progress during the war, the number of workmen employed and the value of the production having increased from 46,412 and £3,860,000 (£1=25 lire) in 1914 to 58,871 and £14,790,133 in 1918, respectively. The chief mining districts are situated in the provinces of Caltanissetta (Sicily), Florence, Arezzo, Grosseto (Tuscany), Cagliari, Sassari, and Iglesias (Sardinia), whilst others of lesser importance are situated in the provinces of Bergamo, Brescia, and Turin. The full extent of the mineral resources of Italy is not known and an extended study is required. Details of the output of minerals and metals for 1918 and 1919 (in metric tons) are appended:—

Minerals:—	1919.		1918.	
	Metric Tons.		Metric Tons.	
Iron ore and manganese iron .. ..	608,116	..	450,729	..
Manganese ores .. ..	1662	..	23,502	..
Copper ores .. ..	89,947	..	16,303	..
Lead ores .. ..	44,054	..	11,937	..
Iron and copper pyrites .. ..	317,334	..	372,474	..
Mercury (metal) .. ..	1004	..	548	..
Sulphur .. ..	406,406	..	226,126	..
Coal and lignite .. ..	701,079	..	1,158,542	..
Petroleum .. ..	6372	..	4851	..
Marble .. ..	509,342	..	265,744	..
Metals:—				
Pig iron .. ..	426,754	..	239,716	..
Iron and steel .. ..	933,500	..	731,823	..
Copper .. ..	2091	..	1243	..
Zinc .. ..	—	..	1232	..
Lead .. ..	21,674	..	16,590	..
Antimony .. ..	76	..	30	..
Aluminium .. ..	874	..	1,673	..

The total capital invested in the Italian mining industry at June 30, 1919, was £13,472,278.—(*Bull. Dept. Tr. and Com., Can.*, Nov. 15, 1920.)

**Dutch Guiana in 1919.**—The production of gold is steadily declining, although dredging prospects in the Marowynne river are considered good. Bauxite mining is regulated by a new law, which provides for concessions lasting not more than 50 years and limits the area held by any one company or individual to 308,750 acres. Preparations for the mining and export of bauxite are going forward rapidly. Mercury has been found in a number of places near the Marowynne River, and in co-operation with the Government a Dutch company is exploring large areas said to contain deposits of high-grade iron ore. Balata production has decreased, in spite of good prices, and as areas worth exploiting are becoming more inaccessible from the coast,

the industry is gradually passing into the hands of two or three large corporations.—(*U.S. Com. Rep., Suppl., Oct. 4, 1920.*)

**The Mica Industry in Brazil.**—H. M. Commercial Secretary at Rio de Janeiro reports that excellent sheet mica is produced in Brazil in the State of Minas Geraes (municipality of Pecanha). Large quantities of good-quality mica also occur in the State of Sao Paulo, in Iguape, Itapeirica, and in the Serra do Mar mountains. The development of the industry was formerly greatly handicapped by inadequate transport, but apparently this is now largely counterbalanced by increased working costs in India and elsewhere, and by the higher price of the product. The production of mica in Brazil has risen from 50·8 metric tons in 1915 to 154·35 t. in 1919.—(*Bd. of Trade J., Dec. 16, 1920.*)

**Proposed Pulp and Paper Manufacture in Portuguese E. Africa.**—H. M. Consul-General at Lourenco Marques reports that a concession has been granted to the Cia. Portuguesa de Papel Mocambique giving it the exclusive right to prepare pulp from native and foreign vegetable matter, and to manufacture paper and parchment in the province of Mozambique for a period of ten years. The company is to erect a factory at Ressano Garcia, near the Transvaal frontier, and utilise tambuki grass, and possibly wood, as raw material. The syndicate which holds the concession for the manufacture of pulp from baobab trees in the Quilimane and Tete districts has been unable to sell its products to paper-manufacturers in the United Kingdom, in spite of favourable technical reports. Some time ago it contracted to supply a Norwegian pulp company with 40,000 tons of material on a royalty basis, and since then it has received numerous inquiries from Germany and the United States.—(*Bd. of Trade J., Dec. 16, 1920.*)

**A New Process for Drying Peat.**—A company, the Oy. Hydroturpe A.B., has been formed in Finland for the purpose of exploiting a new labour-saving process for drying peat. In the process the peat in the hed is reduced to mud by a jet of water under high pressure, and the mud is pumped on to a drying field by an electrically-driven pump fitted with a cutting apparatus. Roots, etc., are removed by means of a special device and, after drying, the mud is cut up into uniform sizes. Peat ("hydro-turf") prepared by this process, which is of Russian origin, is said to dry more quickly than ordinary peat.—(*U.S. Com. Rep., Dec. 30, 1920.*)

**The Institute of Metals.**—The annual general meeting of the Institute of Metals will be held on March 9 and 10 next at the Institution of Mechanical Engineers, Storey's Gate, Westminster. The meeting will commence on each day at 10.30 a.m., and the list of papers to be read includes:—March 9. "Stages in the Re-crystallisation of Aluminium Sheet on Heating, with a Note on the Birth of Crystals in Strained Metals and Alloys": H. C. H. Carpenter and F. Elam. "Some Notes on Calcium": P. H. Brace. "Plastic Deformation of Some Copper Alloys at Elevated Temperatures": C. A. Edwards and A. M. Herbert. "The Action of Reducing Gases on Heated Copper": H. Moore and S. S. Beckinsale. March 10. "The Season Cracking of Brass and other Copper Alloys": H. Moore, S. Beckinsale, and C. E. Mallinson. "The Constitution of the Alloys of Copper with Tin, Parts III. and IV.": J. L. Haughton. The annual dinner will be held at the Trocadero Restaurant on March 9, at 7.30, and on the afternoon of March 10 a visit will be made to the National Physical Laboratory, Teddington. As previously announced, the May Lecture will be given on May 4 by Prof. T. Turner on "The Casting of Metals."

It is announced that arrangements have been

made for the publication of an index to the first twenty volumes of the Institute's Journal, and members are asked to notify the Secretary if they intend to purchase a copy, the price of which will probably be 10s. 6d. The next ballot list of applicants for membership will be issued on February 24, and applications should be sent in by the day before. Special concessions are offered to those who are elected members on the next ballot.

**Lectures on Catalysis at the Royal Society of Arts.**—A series of three Cantor Lectures will be delivered during February by Dr. Eric K. Rideal on "Applications of Catalysis to Industrial Chemistry." The syllabus is as follows:—

Lecture I. Feb. 14.—Introduction: classification of catalytic action: theories of catalysis; technical difficulties.

Lecture II. Feb. 21.—Processes of Oxidation: Sulphuric acid: nitric acid: chlorine: catalysts in the gas industry: sulphur recovery: surface combustion: incandescent mantles: organic industries: formaldehyde: drying oils: linoleum: oxidation of hydrocarbons.

Lecture III. Feb. 28.—Processes of Hydrogenation:—Preparation and purification of hydrogen: methane: hexahydrobenzol: oil-hardening: synthesis of ammonia: the cracking of oils: synthetic rubber. Hydrolytic Processes:—Saponification: glucose: alcohol: acetic acid: acetone and ether.

The lectures begin at 8 p.m.

## LEGAL INTELLIGENCE.

**ALLEGED BREACH OF CONTRACT re SALE OF COAL-TAR PITCH.** *Erlbach and Co. v. Gatty, Saunt and Co.*

In the Court of Appeal on January 21, Messrs Gatty, Saunt and Co. appealed against a judgment of Mr. Justice Bailhache, awarding Messrs. Erlbach and Co. damages for alleged breach of contracts relating to the sale of coal-tar pitch. Plaintiffs (Messrs. Gatty, Saunt and Co.) contended that some of the pitch supplied was not coal-tar pitch, and was unsuitable for briquette manufacture, for which, to the defendants' knowledge, was required, and for which they gave an assurance that it was suitable. Defendants denied having any knowledge that the pitch was required for that purpose stated, or having given any warranty, and did not admit that the pitch was not coal-tar pitch. Mr. Justice Bailhache found for the plaintiffs both points, viz., that the pitch supplied was blast-furnace coal-tar pitch, and was therefore not a delivery; and that plaintiffs were told that the pitch was suitable for briquette manufacture.

In dismissing the appeal, Lord Justice Bankes and Scrutton and Adkin found that, although there was no well-known and accepted meaning of the expression "coal-tar pitch," blast-furnace pitch was excluded from the terms of the contract. Fit that stood high and desired to be beyond reproach would not sell blast-furnace pitch as coal-tar pitch. On the other hand, they were satisfied that plaintiffs had informed defendants that the pitch was required for briquette-making, and this implied warranty. The judgment in plaintiffs' favour therefore stands.

**PUBLICATION OF A DESIGN.** *Gunston, Green Simpson, Ltd., v. Wincox, Ltd.*

In the Court of Appeal, on February 23, Master of the Rolls and Lords Justices Warrington and Younger allowed an appeal by Messrs. Wincox Ltd., from a judgment of Mr. Justice Asth given at a suit of Messrs. Gunston, Green Simpson, printers, of Farringdon Avenue, E.C.

By the judgment complained of Mr. Justice Astbury granted an injunction to restrain the defendants from infringing plaintiffs' design (a card-cut-out portraying a nurse carrying a tray with wine and bunches of grapes). Plaintiffs' case is that, having originated the design and applied for registration, they sold it to Liebig's, Ltd., for advertisement purposes, but that before any copies of the cut-out were delivered they found that defendants were using a design which they considered an infringement. Mr. Justice Astbury held that the manager of Winox, Ltd., having been shown the design in confidence, had something similar prepared to his order from other printers, and granted an injunction.

The Master of the Rolls, giving judgment, said he agreed with Mr. Justice Astbury with regret, because he thought the defendants had played the plaintiffs a very dirty trick. But before the design is shown to the defendants, and before it was registered, it had been shown to another firm with the view of obtaining an order, and that constituted an publication within the meaning of the Patents and Designs Act, and enabled the defendants to escape the consequences of the conduct of their manager. The appeal must be allowed. The Lords Justices concurred.

## REPORT.

REPORT ON THE INDUSTRIAL AND ECONOMIC SITUATION IN GERMANY IN DECEMBER, 1920. By J. W. F. TRELWALL and C. J. KAVANAGH. Pp. 79. Department of Overseas Trade. London: H.M. Stationery Office. 1920. [Cmd. 1114. 9d.]

Although conditions in Germany have generally improved during the past two years, the state of public finance has become desperate owing to the ever-increasing issue of paper currency. In March, 1920, trade and industry were plunged into sudden stagnation, which still continues, although the critical stage is probably past. The high dividends paid for the year 1919 seem to suggest successful trading, but some of the satisfactory balance sheets are due to large payments by the State on completed war contracts. The outstanding features of the past year were the formation of combines and operation with foreign groups; the former movement has spread very widely, especially in the iron, steel, and coal industries. No official statistics of foreign trade have been published. The labour situation, as a whole, has improved, but it may revert in times of stress arising from failures due to losses in exchange consequent on falling world prices, or from increased taxation.

Complaints of the shortage of coal are not directly confirmed by the published figures of production and distribution. Thus it was officially stated that the proportion of supply to requirements in August last, compared with that in August, 1913, was 64 per cent., but the issued statistics indicate 82 per cent. Only 88 per cent. of the coal requirements of the chemical industry stated to have been satisfied in May, and 63 per cent. in August, 1920. Owing to the increased consumption of lignite (now 132 per cent. above that of 1913), alterations have had to be made in firing systems and greater space is required in transport; gas power stations, nitrogen plants, etc., use it exclusively. There have also been improvements in utilisation of waste gases, peat, and other low-grade fuels. To sum up, although German industry is not abundantly supplied with coal, it is by no means threatened with annihilation from lack of it; difficulties are only temporary and coal production is improving.

The iron and steel industry has recovered rapidly and a measure of stability has been attained since

April, 1920, when the Eisenwirtschaftsbund came into operation. As most of the iron ore consumed has now to be imported, attention will be directed more to working up iron and steel into the highest types of manufacture rather than producing semi-finished products, e.g., pig iron, blooms and billets, as in the past. One of the most important combines (*cf. s.*) is the Rhine-Elbe-Union which controls a capital of over 500 million marks and employs 200,000 workers.

The non-ferrous metals industry has re-established itself on old lines. The aluminium industry is now controlled by the Vereinigte Aluminiumwerke A.-G. and has enjoyed a good export trade; home deposits of bauxite have been developed for chemical rather than metallurgical uses, and supplies formerly obtained from France are being derived from Dalmatia.

Little has been published of late concerning the chemical industry, and the dye industry in particular, save complaints about the coal shortage and the high cost of raw materials and labour—difficulties which have led to further amalgamations. Several Silesian firms have combined under the title "Chemische Werke Oberschlesien G.m.b.H.," with the object of working up their total production of tar, benzol, and ammonia. The chemical industry has survived the most critical stage after the war, and helped by its consolidation, organisation and experience, is trying to regain its old markets. The H. J. Sloman Saltpeter A.-G. (*cf. J.*, 1920, 281 R) is to sell its overseas interests to the Salitrera Tocopilla company for 16,320,000 mk., its shareholders having the right to acquire an interest in the new company. In the early part of the year the potash industry improved, crude salts were in plentiful supply, but salts high in potash were scarce owing to exportation and lack of coal. Towards the autumn, the trade with America declined and Alsatian competition began to be felt. The consumption of pure potash (K<sub>2</sub>O) in 1919 was 812,002.4 metric tons compared with 1,110,369.4 t. in 1913, and 1,001,664.3 t. in 1918, and the sales from January to October, 1920, were about 800,000 t. Sales of manufactured potash compounds have notably decreased. Whereas potash prices have risen by an average of only 441 per cent. since 1914, those of coal, iron, food and wages have risen by 1600, 2700, 1200 and 1000 per cent., respectively. As the home price of potash is below cost, extra profit must be made on foreign sales. Since 1913 a potash council (Reichskalirrat) of a socialistic nature has been formed to enable employers, consumers, and workmen to meet and discuss conditions of labour, wages, etc. (*cf. J.*, 1920, 100, 116, 132, 235, 274, 446 R).

The seed-crushing industry was in a serious position in September owing to stocks having become exhausted through official interference. Some German and Dutch firms are forming a company with a capital of 100 million mk. (*cf. J.*, 1920, 445 R) in order to secure adequate supplies of fats and to overcome difficulties of exchange in purchasing raw materials; the purchases will be effected through the Dutch parent undertaking. Of the 102 mills associated with the German Economic Union of Oil-pressing Mills, a large majority is under foreign (mostly Dutch) influence.

An industrial organisation ("Reichswirtschaftsstelle für Kunstseide und Stapelfaser"), in which makers, consumers, and employees have equal representation, was formed in November to regulate production, sales, and prices in the artificial silk industry, and its first action was to abolish State control. The production of artificial silk has so increased that it can now satisfy the home demand.

The report contains fifteen appendices giving statistical information relating to finance, fuel, wages, unemployment, industrial combines, foreign trade, etc.



## OFFICIAL TRADE INTELLIGENCE.

(From the Board of Trade Journal for January 27 and February 3.)

### OPENINGS FOR BRITISH TRADE.

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W. 1, from firms, agents or individuals who desire to represent U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number.

Locality of Firm or Agent.	Materials.	Reference number.
Canada .. ..	Chemicals, dyes, oils, glue, fertilisers, dry colours, whiting, precipitated chalk .. ..	123
" .. ..	Metals, leather, china, porcelain .. ..	154
" .. ..	Bessemer blue-annealed steel sheets .. ..	163
Egypt .. ..	Soap .. ..	163
" .. ..	Paper, bristol-board, ink, antimony, tin, pig lead (tenders for) .. ..	127
South Africa ..	Paper .. ..	132
Belgium .. ..	Paint, varnish .. ..	133
" .. ..	Metallurgical products .. ..	169
Norway .. ..	Pitch .. ..	140
Switzerland ..	Leather .. ..	143
China .. ..	Black and galvanised sheets, tinplate .. ..	174
Persia .. ..	Linseed oil .. ..	176
United States ..	Drugs, soap, window glass .. ..	177
" .. ..	Barytes, lithopone, dry colours, cresylic acid .. ..	178
" .. ..	Mica .. ..	181
" .. ..	Glass, pottery, china, porcelain .. ..	182
" .. ..	Scientific and optical instruments .. ..	183
" .. ..	Earthenware, pottery .. ..	184
" .. ..	Pottery, tiles .. ..	184

\* The High Commissioner for Canada, 19, Victoria Street, London, S.W. 1.

**MARKETS SOUGHT.**—A Canadian firm wishes to get into touch with U.K. importers of cyanide for the extraction of gold. [Inquiries to the Canadian Government Trade Commissioner, 73, Basinghall Street, London, E.C. 2.]

A firm in Ceylon and Southern India able to export medicinal plants, vegetable fibre, copra, rubber and oil-seeds wishes to hear from U.K. importers. [128]

### TARIFF, CUSTOMS, EXCISE.

**Australia.**—Recent customs decisions affect iron and steel sheets, carbonate, bicarbonate and silicate of soda, and explosives.

**Austria.**—Customs duties when paid in bank notes must be paid at 50 times the rates prescribed in the tariff, as from January 1.

**Belgium.**—The conditions affecting the import and export of sugar are given in the issue for January 27.

Cocoa butter, salt, and soap may now be exported without licence.

**British India.**—The duty on opium imported into the Punjab and the North-West Frontier Province is fixed, as from April 1, at 36 Rs. per seer unless produced in any State under the control of the Punjab Government, in which case the duty will be 27 Rs. per seer.

**Colombia.**—Export of gold and silver is now permitted. The customs duties have been modified on, *inter alia*, alcohol, certain alcoholic beverages, and printing paper.

**Denmark.**—Export prohibitions have been withdrawn from oil-cake, margarine, vegetable oils for the manufacture of margarine, oleo stock, oleo-oil, certain oil-seeds and oleaginous products.

**Egypt.**—The export of coal-tar derivatives suitable for the manufacture of explosives is prohibited. The export is prohibited, except under licence, of crude alcohol, groundnuts, silver, butter, certain drugs, chemical fats, edible fats, cotton oil, sesame oil, yeast, gold, crude paraffin, molasses, sugar, and oil-cakes.

**France.**—The customs duties on certain kinds of paper have been reduced.

The prohibition of the export of pure vegetable oils has been withdrawn, but the export of oil-cake is still subject to licence.

**French Equatorial Africa.**—An export duty of 5 per cent. *ad valorem* has been levied on palm oil and palm kernels.

**Italy.**—The customs duty on mineral oils, except heavy oils, has been increased by 50 per cent.

Benzol and other light coal-tar oils for use as raw materials for the manufacture of artificial organic colours, varnishes or lacquers, may be admitted at the reduced rate of 2 lire per 100 kg. if denatured by specified methods.

Ammonium nitrate for use as a fertiliser may be admitted duty free if adulterated and upon proof of its use for this particular purpose.

The temporary suspension of the import duty on white paper, in rolls, for newspapers is extended until March 31.

The use of white phosphorus for the manufacture of matches, and the import of matches containing white phosphorus, are prohibited as from January 1. The import of white phosphorus for other uses is subject to the permission of the Minister of Finance.

The import of motor spirit, matches, saccharin, and sugar is reserved to the State.

Among the articles for which import licences are still required are glassware and perfumery.

The export of the following is prohibited:—Bricks, tiles, cement, charcoal, copra, animal and vegetable fats, feculæ, margarine, condensed milk, fertilisers, metal scrap, certain metallic ores, nickel, newsprint paper, glucose, molasses, sugar, caramel, certain oils, various potash salts, pyrites, quinine, pure spirit, copper sulphate, tanning extracts, toluol, etc.

**Japan.**—The duty-free admission of mineral oils is only applicable to oils to be used for certain specific purposes, e.g., the ceramic and metal-heating industries.

**Memel Territory.**—The customs régime is given in the issue for January 27.

**Netherlands.**—Export prohibitions have been withdrawn from engine and lubricating oil, fire-bricks, coal tar and its residues.

**New Zealand.**—As from December 1, 1920, packages containing cream of tartar substitute must be labelled with the name or names of the substances most generally understood by the public.

The prohibition of the export of sheep skins has been revoked.

**Norway.**—The import of alcohol is prohibited.

**Portugal.**—A revision of the Customs tariff is projected. The proposed new rates are not very different from the tariff of 1892, but the duties are leviable entirely in gold. Payment, however, may be made by means of "gold warrants."

**South Africa.**—Sugar imported from Belgium and Holland is subject to a surtax.

**Spain.**—The rate of surcharge on customs duties when paid in Spanish silver or notes of the Bank of Spain is fixed for the month of February at 45/63 per cent.

**Trinidad and Tobago.**—Articles subject to export tax include sugar, molasses, syrup, rum, cocoa, coconuts and copra.



## COMPANY NEWS.

## GAS LIGHT AND COKE COMPANY.

Speaking at the annual meeting on February 4, Mr. D. Milne Watson, the Governor, announced that the company would shortly sell gas on a calorific basis, and that the standard price would be 1s. 3<sup>2</sup>d. per therm. The calorific value to be declared was 475 B.Th.U., and the actual price would be 1s. 2d. per therm, corresponding to the present price of 5s. 6d. per 1000 cb. ft. of gas. The sliding scale had now been re-established, but on a different basis, and it should be possible to raise the dividend from £3 to £4 13s. 4d. per cent. per annum. Mr. Milne Watson dealt at length with the reasons why the price of gas had recently been raised by 10d. per 1000 cb. ft., the chief of which was the very sharp decline in the price of residuals. He also reviewed in detail the events of the past year, stating that the output of gas had reached the record total of 32 thousand million ch. ft., that the number of customers had increased by 9000, and that the coal consumed had risen to over 2½ million tons. Both the increased demand for gas and the good prices obtained for residuals (until near the end of the year) were satisfactory, but on the other hand the company had suffered from the rise in the cost of coal, the very high price of oil, the enormously increased wages bill, the necessity for spending large sums on repairs and maintenance, and the slump in the price of residuals following the coal strike. The company was extending its manufacture of intermediates in order to meet the new situation created by the passing of the Dyestuffs (Import Regulation) Act.

**AMALGAMATION IN THE PHOTOGRAPHIC TRADE.**—An amalgamation has been arranged between the following firms:—Paget Price Plate Co., Watford; Rajar, Mohberly, Cbeshire; Marion and Co., Soho-square and Southgate; Marion and Foulger, Bedford; A. Kershaw and Son, Leeds; Kershaw Optical Co., Leeds; Rotary Photographic Co. (1917), West Drayton. The new company is called the "Amalgamated Photographic Manufacturers, Ltd.," and its authorised capital is £1,100,000, divided into 100,000 seven per cent. cumulative preference shares of £1 each, 600,000 ten per cent. participating cumulative preference shares of £1 each (250,000 of these are now being offered at par), and 400,000 ordinary shares of £1 each.

**THE BRITISH CELLULOSE AND CHEMICAL MANUFACTURING CO., LTD.**, has received a report from Messrs. Clavel and Lindenmeyer, dyers, of Basle, Switzerland, describing the results of tests on the dyeing properties of the cellulose acetate silk made at Spondon. According to the report, this silk shows a remarkable chemical affinity for dyes, and no pre-treatment is required even in the case of dyes insoluble in water. The Basic, Azo, Acid, Alizarin, and other dyes can be used successfully, and the dyed silk thus obtained is fast to light and wash proof; the basic dyes in particular show an unusual fastness to light. Weaving tests have shown that the great elasticity of the silk enables it to be worked through the loom with ease, whether used as warp and weft, or as one of these, or in conjunction with natural silk; those responsible for these tests remarked on the absence of breakages and called attention to the fine "scroop" and rich appearance of the product.

**BRITISH CYANIDES CO., LTD.**—An extraordinary meeting was held on February 9, at which certain arrangements proposed by the directors relating to the development of the barium cyanide process of fixing atmospheric nitrogen were approved. The

experimental work connected with this process has been done by the Oldbury C.S. Syndicate, an offshoot of British Cyanides Co., Ltd., and the main difficulty of obtaining the right kind of furnace is now believed to be overcome by a new furnace designed by Arthur Duckham and Co. An agreement between this company and the Syndicate is now to be signed which gives the latter, *inter alia*, an exclusive licence to use the patents taken out by Sir Arthur Duckham or his firm relating to furnaces used in the barium or similar processes, in return for a share interest in the Syndicate.

## TRADE NOTES.

## BRITISH.

**Fiji in 1919.**—The chief products of Fiji are sugar, copra, and bananas. Although the sugar industry was suffering from a lack of labour, due to the disorganisation of Indian immigration, the amount of sugar shipped was 64,348 tons, or 1278 t. more than in 1918. Additional areas of coconut trees are coming into bearing, and the export of copra will probably increase steadily; exports in 1918 and 1919 were 19,318 t. and 27,311 t., respectively. The increased export of rubber from 36½ t. in 1918 to 69 t. in 1919 was due to some extent to shipments held over from the previous year; the rubber trees continue to give satisfactory results, large areas are available for cultivation at reasonable rentals, and the prospects of the industry are favourable.—(*Col. Rep.—Ann., No. 1047, Nov., 1920.*)

**Gambia in 1919.**—In the colony of Gambia, which covers 4132 sq. miles, exist many deposits of iron ore, ochre, and china clay, but none of these is mined. The iron ore is situated most favourably for direct loading into ships, but is of poor quality. The staple industry is farming, the chief crop being groundnuts, in which there is a considerable export trade. As an area of 640,000 acres of suitable soil is available for this crop, there is scope for considerable expansion. The collection and export of wild rubber have practically ceased. Small amounts of cotton are grown, but the further development of this and of other commercial crops is severely limited by the scarcity of rain. The exports, valued at £1,553,521, included 70,270 tons of groundnuts and 671 t. of palm kernels; gum and wax have practically disappeared from the export list. Of the import trade, valued at £1,250,320, the United Kingdom supplied 57 per cent., and the United States 19 per cent. (4 per cent. in 1915).—(*Col. Rep.—Ann., No. 1053, Dec., 1920.*)

## FOREIGN.

**Trade of the Belgian Congo.**—The Belgian Congo has developed rapidly since it was opened to world trade, the mining industry shows increased returns every year, and agriculture is making good progress. Coffee and rice are now staple exports; cotton growing is promising well, the 1920 crop being estimated at 982 tons. The export trade in 1917, valued at £7,164,022, showed an increase of over 250 per cent. as compared with 1913, and comprised 3700 long tons of rubber, 33,487 t. of palm oil, and considerable quantities of palm-nuts, cocoa, copal, ivory, coffee, timber, and various minerals. The imports, valued at £2,767,239, were derived chiefly from the United Kingdom, South Africa and Rhodesia, followed by the United States and France, the share of Belgium being small owing to export restriction. Among the imports were:—Cement, 2699 t.; petroleum, gasoline, etc., 2696 t.; colours, dyes, and varnish, 240 t.; and soap, 534 t.—(*U.S. Com. Rep., Nov. 4, 1920.*)

**Portuguese Trade in 1919.**—Although the export trade in 1919 increased but slightly, the value of the imports more than doubled, owing to diminished production, the introduction of the 8-hour day, and the need for replenishing stocks. Business, generally, was prosperous, coal again became available, and the cork industry had a good season.

The imports, valued at £38,600,500 (escudo = 3s. 4d. at par), included:—Cement, 12,155 metric tons; sodium borate, 44 t.; sodium carbonate, 1289 t.; sodium sulphate, 280 t.; caustic alkali, 2423 t.; carbonate of calcium, 1233 t.; chlorate of calcium, 1038 t.; nitrate of potash, 142 t.; nitrate of soda, 218 t.; quinine salts, 70 t.; copper sulphate, 2043 t.; other chemicals and drugs, 4844 t.; coal, coke, etc., 609,363 t.; dyes and colours, 851 t.; dyeing extracts, 947 t.; dyewoods, 165 t.; fertilisers, 23,292 t.; oils and fats, animal, 1272 t.; palm oil, 1532 t.; other oils, 322 t.; perfumery, 74 t.; and sulphur, 6305 t. The greater part of the chemical imports is supplied by England.

Among the exports, the par value of which was £18,425,666, were:—Argols, 11,066 metric tons; chemical products, 1526 t.; cork and cork products, 71,223 t.; hides, 1612 t.; cod oil, 524 t.; mineral oil, 569 t.; olive oil, 720,360 litres; copper ore, 18,403 t.; iron ore, 7375; wolfram, 770 t.; other ores, 1101 t.; paper pulp, 858 t.; rosin, 1661 t.; turpentine, 370 t.; wax, raw, 112 t.; vinegar, 320,610 litres; glassware, 365 t.; and soap, 189 t.—(*U.S. Com. Rep.*, Nov. 3, 1920.)

## GOVERNMENT ORDERS AND NOTICES.

**PROHIBITED EXPORTS.**—The following have been removed from lists "A" and "B" of prohibited exports as from February 1:—(1) Coal tar, all products and derivatives thereof, whether actually so obtained or so derived from other sources (including all mixtures and preparations containing such products and derivatives), suitable for use in the manufacture of dyes or explosives; (2) dyes and dyestuffs manufactured from coal-tar products and articles containing such dyes and dyestuffs; and (3) indigo, synthetic.

Yeast was removed from the same lists on January 13.

**DECONTROL OF COAL.**—The Directions of the Board of Trade, dated August 25, 1920, regarding pithead prices of coal, are to be cancelled as from March 1 next, as there is now no danger of shortage within the country, and the export price has approached the home price so closely that there is no longer any reason for restricting prices. Further, the directions for the supply of specified districts or consumers, will cease to have effect from the same date, and inland consumers will then be free to purchase from any source. The position as to the supply of coal for foreign bunkers and exports remains as published in the *Journal of the Board of Trade* for December 28, 1920, and January 10, 1921.

**FIRE PREVENTION.**—A Royal Commission has been appointed to inquire into the existing provision for the avoidance and extinction of fires.

**MINERS' LAMPS COMMITTEE.**—The Secretary for Mines has reappointed, with extended terms of reference, the Miners' Lamps Committee which was appointed by the Home Secretary in May, 1919. The Committee has to report upon possible improvements in the safety and illuminating power of miners' lamps, testing methods, maintenance of the lamps, use in mines which show no indication of inflammable noxious gases, etc. Mr. W. Walker, of the Mines Department, is chairman, and Prof. R. V. Wheeler is a member of the Committee.

## REVIEWS.

**NUCLEIC ACIDS: THEIR CHEMICAL PROPERTIES AND PHYSIOLOGICAL CONDUCT.** By WALTER JONES. Second edition. Pp. viii. + 150. (London: Longmans, Green and Co. 1920.) Price 9s. net.

As the science of biochemistry advances the problem of the nature of the substances most intimately connected with the living cell—which constitute, as it were, the chemical basis of life—is being gradually unravelled and the substances themselves isolated and relegated to their proper position in chemical classification. Within comparatively recent years the sugars, the purines, the proteins, and the lipins have yielded many, though by no means all, of their secrets to the investigator, and some beginning has been made in the study of their functions in the living organism. In the present work an account is given of the elucidation of the chemical nature of another group of substances of great physiological interest—the nucleic acids. The fundamental importance of the nucleus in all the physiological relations of the cell has long been recognised by biologists and renders the story of the discovery of the chemical nature of the nuclear constituents, as told in the first half of Prof. Jones's book, one of intense interest and fascination. The nucleic acids occur in the cell nuclei combined either with proteins, *α-nucleoproteins*, or with the much simpler proteins, in which form they are present, for example, in the spermatozoa of fish. The *α-nucleoproteins* consist most probably of various salts of proteins with nucleic acid, in which the protein is in excess. When they are digested with pepsin part of the protein is digested away, leaving a mixture of nucleic acid salts, indefinitely called *nuclein*. To use the term introduced by Kossel, the nucleic acids for the prosthetic group of the *α-nucleoproteins* are *nucleins*.

Many chemists have contributed to the discovery of the constitution of the nucleic acids, prominent among whom are Kossel, Levene, and Jones, the author of the present work. So far as is known only two nucleic acids exist: that prepared from the cells of animals, generally known as *thymus nucleic acid*; and that obtained from vegetable cells, usually termed *yeast nucleic acid*. These two agree in their general structure but differ in the nature of their components and to some extent in their behaviour towards reagents. Yeast nucleic acid, the better known of the two, is resolved by mild alkaline hydrolysis into four groups, known as *nucleotides*, each of which is a compound of phosphoric acid, *D*-ribose and a base, probably combined in that order. Two of these bases are purine, guanine and adenine; the other two pyrimidine, cytosine and uracil. These nucleotides are themselves decomposed by further hydrolysis with loss of phosphoric acid and formation of *nucleosides*, each composed of *D*-ribose and the corresponding base:—

	Nucleotides.	Nucleosides.
Yeast nucleic acid →	Guanine nucleotide	→ Guanosine
	Adenine "	→ Adenosine
	Cytosine "	→ Cytidine
	Uracil "	→ Uridine

The exact mode of combination of the various components within the nucleotides and of the nucleotides with each other is not yet definitely settled.

Thymus nucleic acid is probably constituted after a similar plan, but the sugar group is a hexose, and the four bases are guanine, adenine, cytosine, and thymine.

The second part of the book is devoted to the less fascinating story of the physiological conduct

the nucleic acids, which affords an admirable example of the remarkable complexity of chemical changes in the living organism as well as of the extraordinarily specific adaptation of its enzymes to the materials upon which they have to exert their action. Briefly put, nucleic acid is first decomposed into its constituent nucleotides by means of *nucleinase*, present in the intestinal juice. The nucleotides thus liberated may undergo decomposition in either of two ways. First, by the action of a *phospho-nuclease*, they may yield phosphoric acid and the corresponding nucleoside. This is the only change observed with the two pyrimidine nucleosides, and the fate of the resulting pyrimidine nucleosides is not fully understood. The purine nucleosides, however, may also be acted on by a *phospho-nuclease*, yielding a purine and a carboxate phosphoric ester. The purine nucleosides also be directly hydrolysed, by a *nucleosidase*, into a purine and a carboxylate. Here, however, a further complication arises, for the purine bases themselves and the purine nucleosides may be deaminated (by specific enzyme action), guanine yielding xanthine, and adenine, hypoxanthine, whilst the nucleosides undergo a similar change, forming nucleosides, which on hydrolysis (again by the same action) yield xanthine and hypoxanthine. Finally, oxidising enzymes come into play, and the xanthine passes first into xanthine and then uric acid. Here in man and the higher apes the process stops and the uric acid is excreted, or is in the system to cause those ills which are the bane of our evolutionary superiority. In the lower mammals, including the monkey, the uric acid is most completely oxidised to allantoin, which is excreted.

For the biochemist this book cannot fail to be of great interest, alike for the importance of the subject and the lucidity of the exposition. Appendices concerning practical methods and a bibliography of over five hundred papers add to its usefulness.

A. HARDEN.

ZWISCHENPRODUKTE DER TEERFARBENFABRIKATION. By OTTO LANGE. Pp. xxiv. + 645. Leipzig: Otto Spamer. 1920. Prices:—*Unbound, Germany*, 135 marks plus 40%; *England and Colonies*, 80s.

So closely bound up with the vitally important problem of developing our industry in fine chemicals, including the coal-tar intermediates, is the question of the provision of a technical literature of an informative character. Although the production of such treatises is more prevalent now than formerly in this country, English-speaking chemists are still largely dependent on German books for the detailed information which is vital to a working specialist in any of the main branches of chemical technology.

The appearance of the volume under review is full of many evident signs that this hegemony in technical literature will not be easily surrendered, whatever developments the future may bring, an indisputable fact that the published records of progress achieved in coal-tar chemistry during the last two or three decades are to be found largely in German patent specifications.

The author introduces his subject by laying stress on the circumstances that the coal-tar intermediates form the basis of the synthetic colour industry. The task he has set himself is to present within a moderate compass, in tabular form, an epitome of a very large group of chemical substances. The results of the work may be gauged by noting that his tabulation contains 3637 entries; but these are sent a far greater number of compounds, for numerous instances the entry is generic and conveys references to many chemical entities. The whole is in the first instance divided into four

principal sections under the headings of benzene, naphthalene, anthracene, and phenanthrene, and in each of these sections the derivatives of the parent hydrocarbon are arranged in increasing order of complexity. The introduction and summary of contents furnish an explanation of the system of arrangement whereby it is easy to find the reference to any desired intermediate. Each reference contains a characteristic number by means of which the intermediate or group of intermediates can be located on looking up the comprehensive index. The patent is cited in which the given intermediate was first described, and this quotation is followed by a concise summary of the method of preparation.

In the vast majority of cases references are given only to German patents, but occasionally English, French, and American specifications are quoted, but only as subsidiary citations. In many instances this information is supplemented by references to German scientific journals. Providing that the reader has access to the principal German patents on coal-tar intermediates, such as those reproduced in Friedländer's comprehensive treatises on the progress of the coal-tar industry, then the problem of tracking down any particular intermediate will be greatly facilitated. From the number of entries it is obvious that a very large number of intermediates are tabulated, but at the same time many substances of purely scientific interest are omitted, since these can be found by reference to the lexicons of Richter or Stelzner. Considerable space is saved by omitting all names of authors or patentees. On the other hand, the parts by weight of the reagents, the physical constants (melting and boiling points) of the products, and other experimental details are indicated whenever these are likely to be of practical utility.

Cross references are furnished by the addition of lists of the German provisional and complete specifications (Anmeldungen and D.-R.P.P.), so that when a patent number is known the compound or compounds described in the specification can be identified.

The text is remarkably free from typographical errors and other inaccuracies, and the entire treatise is a simple guide to the more detailed information scattered through German patent literature.

G. T. MORGAN.

BENZOL: ITS RECOVERY, RECTIFICATION, AND USES. By S. E. WHITEHEAD. With an introductory note by the Rt. Hon. LORD MOUNTBATTEN. Pp. xiv. + 209. (London: Benn Bros., Ltd. 1920.) Price 12s 6d net.

Although the recovery of benzene and its homologues from the coal gas produced in coke ovens has been practised on the large scale for many years, the literature on the subject is very scattered and incomplete. The demand arising out of the war for the maximum production of benzene and toluene as the main raw material for high explosives, rendered necessary the equipment not only of all coke-oven installations with "benzol" plant, but also, as far as practicable, the adoption of the process in gas works; and in carrying out this work the want of a book dealing with the subject was seriously felt. This want still continues in spite of the decreased requirements for explosives, as the combined demand for these hydrocarbons in the organic chemical industry and as motor spirit is far in excess of the supply.

Mr. Whitehead's book is designed to fill the gap and to incorporate the results of the author's experience on the technical staff of the Department of Explosives Supply during the war, with a very large number of installations of "benzol" plant, varying from fully equipped large-scale plants to small emergency plants often constructed largely

the Journal form a fund of information in condensed form, but they are naturally of a somewhat disjointed character, and it is not easy for the reader to gather from them a connected account of what has been or is being done in branches of industry other than his own. For this reason the Annual Reports should prove an indispensable addition to the library of every chemist and manufacturer, and it is hoped that those who have realised their value will help in making their character more widely known among those who have not.

In the space available here it is impossible to give a complete account of the contents of the volume now in the press, but a few notes on some of the items dealt with may be of interest.

In the section on "Plant and Machinery," Dr. W. B. Davidson gives an account of the new electrostatic dust separator erected at Skinningrove. The subject of lubrication is also fully dealt with both in this section and in that on "Mineral Oils" (*cf. i.*). He also deals with the ever-important question of efficiency of combustion and fuel conservation, subjects which are also treated from their respective standpoints by Prof. J. W. Cobb and Mr. H. J. Hodsman in the section on "Fuel" and by Dr. G. Weyman in the section on "Gas and Carbonisation." In these two sections also the Gas Regulation Act receives due attention, whilst the reports indicate distinct, though slow progress in the elucidation of the chemical nature of coal and the mechanism of combustion, and the improvement of carbonisation. The work of the Nitrogen Products Committee, as revealed in its report published early last year, is alluded to in these and in several other sections of the volume, where it touches on the heavy chemical, electrochemical fertiliser and other industries. Dr. A. E. Dunstan, in the section on "Mineral Oils," discusses the conservation of oil resources and the best methods of utilising available supplies; he also deals, *inter alia*, with the conversion of paraffins into fatty acids (a topic also discussed by Mr. Allan), the manufacture of helium, and the progress of "colloidal" fuel.

In the section on "Colouring Matters and Dyes," by Mr. F. M. Rowe, there is an interesting account of the commercial development of the industry; in regard to technical progress perhaps the most interesting work of the year, from the standpoint of the general reader, is that dealing with photosensitising dyes—a subject which is naturally also considered fully in the photographic section. Mr. S. S. Napper records a notable improvement in the character of the work on "Fibres, Textiles, Cellulose, and Paper," especially as regards researches on the chemistry and constitution of cellulose and its derivatives, and on the chemistry and analysis of woods. In the section on "Bleaching, Dyeing, Printing, and Finishing," by Mr. B. Leech, considerable progress is recorded in such matters as the scouring of textiles and the production of Aniline Black.

The most important work of the year in the domain of "Acids, Alkalis, and Salts," dealt with by Dr. T. Ewan, relates to the synthesis of ammonia (with especial reference to the Claude process), the production of nitric acid and of cyanides.

Prof. W. E. S. Turner has much useful work on "Glass" to put on record, especially in regard to optical glass and the effect of varying composition on the properties of glass; it is gratifying to note that much of this work was done in this country. The same author deals with "Refractories," and in this and the section on "Ceramics," by Mr. W. C. Hancock, the predominance of the scientific work done in this country and in America is apparent.

Prof. J. H. Andrew, dealing with "Iron and Steel," remarks that these industries are rapidly becoming more scientific in character. Valuable

work is recorded in regard to blast-furnace practice, special steels, etc., particular note being made of the Report of the Steel Research Committee on the subject of wrought automobile steels.

Prof. C. H. Desch also has useful progress to record in "Metallurgy of the Non-Ferrous Metals"; in this report the flotation process and the metallurgy of zinc and tungsten occupy important places, and full consideration is given to the question of brasses and the theory of corrosion. Reporting on the "Electrochemical and Allied Industries," Mr. D. F. Campbell notes the depressing effects on these industries of the prevailing industrial and financial conditions; much of this section is devoted to iron, the electrical production of steel, and ferro-alloys, electrolytic zinc, and carbon electrodes.

Mr. John Allan comments on the lack of work on the general chemistry of "Oils, Fats, and Waxes." In addition to dealing with the oxidation of paraffins, he also has considerable progress to record on the chemistry of marine animal oils and drying oils, and on the important subject of fat-hardening. Drying oils are also dealt with by Mr. A. de Waele in the section on "Paints, Pigments, Varnishes, and Resins"; in these industries a large volume of work is evident, especially on the examination of pigments and varnish products. Dr. H. P. Stevens records considerable progress in work on the preparation of raw rubber (particularly that carried out in Java) and also on accelerators of vulcanisation, much of this latter being of British origin; the Peachey process of cold vulcanisation is also dealt with. In the section on "Leather and Glue" Mr. F. C. Thompson gives especial prominence to the work on analysis of tanning materials (with special reference to the question of "true tanning value"), the theory of tanning, and the chemistry of hide and gelatin—subjects on which much useful research has been published.

Dealing with "Soils and Fertilisers," Dr. E. J. Russell gives an interesting summary of the nitrogenous fertilisers and deals adequately with other sources of plant food; he also supplies a useful account of recent work on the determination of hydrogen-ion concentration and its significance in agriculture. Mr. J. P. Ogilvie, in the section on "Sugars, Starches, and Gums," traces the progress of the raw material (both cane and beet) through the various processes of sugar making, and deals with the improvements in extraction, clarification (including the use of decolorising carbons), and boiling to grain; the work carried out by the U.S. Bureau of Standards on the determination of sucrose by double polarisation is fully described. Messrs. A. Tait and L. Fletcher contribute the section on "Fermentation Industries," and have much work to record, including a number of papers on the chemistry of enzymes and the fermentation process, yeast, and power alcohol; the work on fermentation acetone is also noted and also that on vitamins in so far as it applies to the industries in question. This latter topic is fully discussed by Dr. H. W. Bywaters in the section on "Foods," and considerable progress in this direction is recorded; much useful work on bread and flour is also noted, and due consideration is given to the report of the Food Investigation Board. Dr. H. T. Calvert deals with "Sanitation and Water Purification," and gives an account of the steps being taken to prevent rivers pollution and the progress towards an understanding of the "activated sludge" process of sewage treatment.

In the section on "Fine Chemicals, Medicinal Substances, and Essential Oils" Dr. T. A. Henry gives instances of the great fall in prices of these products during the last few years. Among the topics with which he deals are the chemistry of cinchona and opium alkaloids, local anaesthetics, essential oils, and organic compounds of metals.

(arsenic, antimony, mercury, silver). Mr. R. E. Crowther contributes the section on "Photographic Materials and Processes," and has considerable progress to record in work on the chemistry of development processes and the preparation of photographic emulsions sensitive to light of all colours; the allied subject of photo-sensitising dyes naturally finds a place here, and some important work on sensitometry is also described.

Mr. W. R. Rintoul has a large mass of work to report in "Explosives," both as regards high explosives for military and for civil purposes, and on the initiation of explosions. Much of the work carried out during the war on nitroglycerin and nitrocellulose has now become available and is duly noted. In conclusion, Mr. C. A. Mitchell has made a judicious selection of the more important literature on "Analytical Chemistry" as applied to technical work in those subjects in the reports of which it has not been dealt with.

The above brief notes may serve to give some idea of the vast amount of work covered by the reports. It will also indicate that there is a certain amount of duplication in the work discussed in the various sections. This is a desirable feature, since it presents important subjects from quite different standpoints—often from those of producer and user respectively—and so enables the reader to gain a more balanced view of the subject.

#### FEBRUARY MEETING OF COUNCIL.

The monthly meeting of the Council was held on February 11, Sir Wm. J. Pope presiding. Among other matters considered the following may be mentioned:—

It was reported that Monsieur Paul Kestner's paper which is to be read at a joint meeting of this Society and the Institution of Mechanical Engineers on March 4 will be printed in the *Proceedings* of the Institution, and that arrangements had been made for the Society to publish in the Journal as much of the paper and discussion as it considers desirable. A supply of advance proofs of the paper will be available for distribution to any members of the Society who may wish to have a copy.

The Council agreed to make a grant of £100, as was done last year, to the Chemical Society towards the expenses of its Library scheme.

Owing to the considerations that a re-transfer of the Society's investments would entail expense, and that a trustee would be ineligible for election to the Council, it was resolved that the investments forming the Messel Fund should be held directly by the Society and not by trustees.

It was intimated that no essay had been received in competition for the Cross and Bevan Prize and the Latham Fellowship, and an alternative scheme was submitted by Mr. C. F. Cross, which was approved by the Council. Particulars of this will be published in due course.

Information was furnished regarding the sailings of steamships to Montreal in time for the Annual Meeting in the Autumn, full particulars of which appear on page 592 of this issue.

Sixty-eight new members were elected, of whom 10 are home members, 2 colonial, 3 Indian, 9 in the United States, and 2 in other foreign countries.

Sir William Pearce, M.P., was elected the Society's representative on the Governing Board of the Imperial College of Science and Technology, in succession to Dr. C. C. Carpenter, resigned.

The Secretary laid on the table the original Register of the Society, which had been discovered during some alterations in the premises of the Alkali Co., Ltd., Widnes.

#### NEWS FROM THE SECTIONS. BIRMINGHAM.

A meeting was held in the University Buildings on January 27, with Dr. H. W. Brownsdon in the chair. Messrs. E. C. Rossiter and P. H. Sanders presented a paper on "The Preparation of Zirconia from Brazilian Ore and a New Method of Determination," in which they described a process for preparing zirconia of 98–99 per cent. purity by fusing the ore with its own weight of caustic soda at a maximum temperature of 600° C., and then proceeding as follows:—The melt is lixiviated with water, washed, and the residue evaporated to dryness with hydrochloric acid. It is then extracted with water and filtered, when 80 per cent. of the zirconia is obtained in solution. The dilute solution is heated with sulphurous acid to reduce the iron salts, and then the addition of dilute sulphuric acid effects a quantitative precipitation of a basic zirconium sulphate having the composition  $5ZrO_3 \cdot 2SO_3 \cdot cH_2O$ . This is washed by decantation, filtered, dried, and converted into zirconia by ignition. The conditions under which the precipitation of the basic zirconium sulphate is quantitative were given.

On February 10, Capt. B. J. Eaton gave a lecture on "Plantation Para Rubber" to a joint meeting of the Section and the Birmingham University Chemical Society.

The lecturer opened with some statistical information, stating, *inter alia*, that of the 350,000 tons of Hevea rubber now produced annually, about one-half comes from British Malaya (where the planted area is about  $1\frac{1}{2}$  million acres), one-quarter from Ceylon, Southern India and Burma, and almost all the remainder from the Dutch East Indies; about 75 per cent. of the world's output is supplied by the British Empire. The production of fine hard Para rubber from the wild Hevea in South America remains stationary at about 30,000 t. per annum, but African and other wild rubbers have practically disappeared from the market. The rest of the lecture dealt with the collection and coagulation of the latex and with chemical problems connected with the preparation of the raw material and the influence of method of preparation and other factors upon the raw product during vulcanisation. At the present time acetic acid is almost exclusively used as coagulant, although, owing to its high cost, it was to some extent replaced by sulphuric acid during the war; but mineral acids are in general unsuitable owing to their influence on the raw rubber during vulcanisation. The use of alum for this purpose in Malaya has been prohibited by legislation. After describing the preparation of smoked and unsmoked sheet and of pale crêpe, and indicating how the mode of preparation influenced the speed of vulcanisation, Mr. Eaton described briefly the work which had been done in the laboratories of the F.M.S. Department of Agriculture, showing how the cause of this variation in speed had been elucidated and how it could be overcome. Finally, in referring to variability in tensile properties, he stated that there was evidence that inferior tensile strength was in some cases due to intrinsic differences in the raw product, i.e., in the caoutchouc, not in the mode of preparation.

#### BRISTOL AND SOUTH WALES.

At an ordinary meeting held at University College, Cardiff, on February 4, Prof. C. M. Thompson presiding, a paper was read by Dr. R. W. Atkinson on "The Iron Ores of South Wales."

After referring briefly to the ironstones of the coal measures, the author proceeded to describe the chief occurrences of iron ore in South Wales. The so-called limonite deposit extends from Rudry to Bridgend and probably further, as there is evidence

of the mineral as far west as Mumbles. The ore occurs between a dolomitic conglomerate and underlying carboniferous limestone; the angle of dip is  $32^{\circ}$  N., frequently following cracks and joints in the strata. Although ore was formerly raised in large quantities from Treacastle Mwyndy, Garth, and Llanharry, to-day the only mine working is that of Llanharry. Analyses show that the preponderating mineral is not limonite, but goëthite ( $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ), the observed ratio of  $\text{Fe}_2\text{O}_3$  to  $\text{H}_2\text{O}$  being very nearly 1:1. Some of the ore very closely resembles the well-known Cumberland kidney-ore in appearance and is also found to be goëthite. Regarding the origin of these deposits, the author expressed the view that the replacement followed the entrance of the iron from above and not from below, as has been thought by some. Experiment shows a marked difference in bulk between the freshly precipitated oxide and the mineral of lesser hydration.

#### NOTTINGHAM.

At a meeting held on February 16, Mr. J. H. Dunford presiding, Sir William Pope gave an address on the scientific aspects of colour photography (cf. J., 1920, 369 n), at the close of which Mr. H. D. Richmond described his early experiments on the same subject.

### FATS AND FATTY ACIDS FROM HYDROCARBONS.

*Synthetic fatty acids.*—Methods for producing fatty acids may be broadly classified into two main groups, in the first of which the fatty acids are built up from hydrocarbons of low molecular weight, such as ethylene or acetylene, or are truly synthesised from hydrocarbons of high molecular weight by means of known reactions. In the second class a hydrocarbon of a higher molecular weight than that of the fatty acid required is submitted simultaneously to partial degradation and oxidation. In this class paraffin wax or high-boiling natural hydrocarbons are obviously available as raw materials.

*Synthesis from compounds of low molecular weight.*—It is not the writer's purpose to describe fully the various methods by which fatty acids have been built up from such substances as ethylene or acetylene. It will be sufficient to indicate the methods of Harries and Willstätter, who propose to synthesise glycerol, and by polymerisation and oxidation to convert it into such acids as oleic acid. When it is considered that ethylene or acetylene is to be converted into formaldehyde or ethyl alcohol, and glycerol obtained from the latter, it will be obvious that the number and complexity of the reactions involved are incompatible with technical and competitive production. The failure of the synthetic-alcohol process in Switzerland—by which calcium carbide was treated to produce acetylene, acetaldehyde produced from the latter by catalytic hydration in the presence of mercuric salts in acid solution, and ethyl alcohol obtained by the hydrogenation of the acetaldehyde in the presence of reduced nickel as catalyst—is distinctly significant in this connexion, for cheap electrical power for the manufacture of calcium carbide and for the production of electrolytic hydrogen, is available in Switzerland, and alcohol prices seem to have been sufficiently high of late to render the prospects of such a synthetic process more than usually favourable.

Further, by assuming an impossibility, namely, that all the complex reactions could be carried out at almost 100 per cent. efficiency, it is clear that,

as all the carbon of the resulting fatty acid must come from the original ethylene or acetylene, a ton of fatty acid could not be produced from less than about a ton of hydrocarbon gas. The production of a ton of the comparatively cheap acetylene necessitates the usage of about four tons of 80 per cent. calcium carbide. Bearing this figure in mind, and making allowances for losses in processes, working expenses, chemicals, power and labour, it is scarcely necessary to elaborate any destructive criticism of processes which use the available low-molecular-weight compounds as starting material for the synthesis of fatty acids.

*The Zelinsky Process.*—The Zelinsky process, by which a paraffin hydrocarbon is chlorinated in the  $\alpha$ -position, a magnesium-alkyl chloride made, carbon dioxide passed in, and the resulting compound acidified to liberate the fatty acid corresponding to the original paraffin, is extremely interesting theoretically, and represents as skilful a piece of work as any of the German synthetic processes. It fails absolutely when subjected to the test of practical applicability, although it is rumoured that the United Chemical Works of Charlottenburg carried it out successfully during the later years of the war. There are, however, several theoretical objections to this process, apart from the cost in metallic magnesium (about 10 per cent. of the weight of fatty acid produced being required) and the cost of chlorine for the original chlorination—the hydrochloric acid produced being used in the final acidification of the "Grignard" carbon-dioxide addition product. In the first place, a satisfactory and determinate chlorination of even an extremely carefully fractionated "cut" of petroleum is apparently impossible. It has been shown by Mahery (J. Amer. Chem. Soc., 28, 165) that a monochloride having the chlorine atom in the  $\alpha$ -position is extremely difficult to prepare.

Again, assuming that a suitable chlorine derivative or derivatives of one or more hydrocarbons of desirable molecular weight could be obtained, the formation of the magnesium-alkyl chloride is not easy. Anyone familiar with the Grignard reaction knows the difficulty of preparing magnesium-alkyl halides from halogen derivatives of hydrocarbons of even moderately high molecular weight. A suitable solvent has still to be discovered in which to carry out the reaction with compounds prepared from high-boiling hydrocarbons. Moreover, the magnesium-alkyl halides of high molecular weight do not react in the same simple direct way as those of lower molecular weight. There is always a tendency to react with the alkyl chloride, with the ultimate production of condensation products of complex hydrocarbons.

On the whole, therefore, it is exceedingly difficult to see how such a complex series of reactions can be carried out on a commercial scale. Zelinsky, it is true, states that satisfactory yields of an acid,  $\text{C}_{18}\text{H}_{36}\text{O}_2$ , were obtained when a hydrocarbon mixture boiling between  $132^{\circ}$  and  $144^{\circ}$  C. was used as starting material, but the results have not been substantiated on a technical scale, and yields would undoubtedly be still lower when higher hydrocarbons were employed with the intention of obtaining acids of the order of stearic and oleic.

There can be no doubt that the Zelinsky process is uneconomical for practical purposes at present.

*The oxidation of hydrocarbons.*—The higher hydrocarbons which occur in the natural petroleum and in the products of destructive distillation of coal, lignite, shale, etc., provide a very attractive and comparatively cheap starting material for the production of fatty acids by oxidation processes. Both paraffins and olefines have been utilised in the suggested processes, and in many instances a relatively expensive paraffin wax has been employed. It is clear, however, that a process, successful when the fairly simple mixture of hydro-



carbons contained in paraffin wax is used as starting material, would not necessarily be applicable to the many varieties of natural petroleum, and this fact should be borne in mind when such apparently satisfactory results as are quoted by Grün (*cf. i.*) are being considered.

The Harries process, in which the high unsaturated hydrocarbons are treated with ozone, the resulting ozonides decomposed by steam into peroxides, and the latter transformed into alkali salts of the higher fatty acids by treatment with caustic alkali solutions, requires little consideration here, although large scale trials have been made. The constitution of natural hydrocarbons, or of those obtained by destructive distillation processes, is by no means clear, but we are sufficiently familiar with its general outlines to know that the formation of an ozonide mixture in appreciable quantity from such oils is scarcely possible. Indeed, Harries states that only 0.12 tons of fatty acids can be produced from the olefines contained in the oils resulting from the destructive distillation of 100 tons of lignite.

The power charges for the production of the necessary amount of ozone are far too high to enable a process dependent primarily upon ozone to be economically feasible when the end product is a fatty acid, and not a "fine" chemical. We may here contrast the production of vanillin from iso-eugenol by means of an oxidation process depending upon the use of ozone. In this case the value of the product allows of a relatively expensive oxidising agent being used, especially as much higher yields result than are obtained by the employment of such oxidising mixtures as potassium bichromate and sulphuric acid.

The direct oxidation of a hydrocarbon mixture, *e.g.*, paraffin wax or a petroleum distillate, by means of atmospheric oxygen offers an attractive solution of the problem of the production of fatty acids. Intense interest has been concentrated upon this question in Germany, and this has led to many superficial attempts to solve the problem by "hit or miss" experimentation. It is true that results have been quoted which appear to be satisfactory, and, within certain limits, the production of fatty acids seems to have been achieved. The names of Franck, Farién, Kelher, Fischer and Schneider, and Löfl, come into mind in this connexion. But one general criticism is suggested here. The results are, as a whole, far too indeterminate, and give little feeling of scientific confidence. We have the so-called influences of this and that catalyst described. We have the effect of pressure and temperature upon the formation of fatty acids discussed, but little valid experimentation is given as backing. We have yields quoted which should assure the commercial success of the processes, and yet fatty acids are not being manufactured. Finally, most of the oxidation experiments were carried out with a high-class paraffin wax as starting material. The hydrocarbons of the latter, under the influence of temperature, pressure and catalyst and with the passage of a current of air, are pyrolytically decomposed and presumably oxidised at the double bond formed on the "splitting" or "cracking" of the hydrocarbons. Even in this case, as is to be expected, a large variety of acids is produced, and this from a fairly simple mixture of hydrocarbons. The difficulty of applying the reaction generally without extensive research upon the various natural oil mixtures is apparent.

Some of the more reliable results, however, indicate a very promising outlook for this process, since the raw materials are cheap, abundant, and available, and the working expenses not, *prima facie*, excessive.

It is known, of course, that the D. Fanto works at Pardubitz have been manufacturing an oxidation

product of paraffin wax for some time, and have been obtaining a mixture of fatty acids which are said to be technically useful. The Franck process, moreover, in which paraffin is oxidised under pressure and in the presence of a catalyst consisting of a resinate of vanadium, manganese, etc., claims to obtain a yield of 85 per cent. of fatty acids, and a net yield of 70–75 per cent. of higher acids suitable for soap-making, esterification to produce fats, and for other purposes.

The results of many researches which have been communicated to scientific societies, especially in Germany, have been exceedingly contradictory, agreeing in practically one thing only, namely, the excellence of the yield of valuable fatty acids.

It is perhaps significant, too, that when the less highly-refined mixture of hydrocarbons resulting from paraffin-wax pressing was employed in place of paraffin wax itself, much poorer yields were obtained in at least one instance.

It is clear from even a cursory examination of the available literature on the subject of the oxidation of hydrocarbons to fatty acids that a great deal of fundamental knowledge is lacking, and that comprehensive researches must be initiated if success is to be attained.

The work of Grün, Ulbrich and Wirth (Ber., 53, 1920, pp. 987–996) appears to afford a striking contrast in many respects to that of several other investigators. It is claimed that the divergent results quoted by different observers are due, in many cases, to the lack of sufficiently accurate analytical control. It is, moreover, preferable to effect a less complete transformation of the original hydrocarbon in order to obtain larger yields of the higher fatty acids. An almost complete conversion often leads to excessive breakdown of the hydrocarbon chain, one carbon atom after another being oxidised away, with the production of lower fatty acids, soluble in water, and technically not so valuable as those of higher molecular weight. It is pointed out that the oxidation can be effected not only with oxygen and air, but also with a gas mixture containing as little as 1 per cent. of oxygen; and under mild conditions of oxidation, and with this 1 per cent. oxygen mixture, only 5 per cent. of water-soluble fatty acids was obtained, whilst simultaneously 50 per cent. of higher, water-insoluble fatty acids was produced. Again, under otherwise suitable conditions, the presence of many so-called catalysts is superfluous—as Löfl has also pointed out—and sometimes actually harmful.

When pure oxygen is used, the reaction is frequently so vigorous as to cause explosions, due to the rapid decomposition of substances of a peroxide nature. In a "normal" oxidation only small quantities of these peroxide substances are produced, and are immediately decomposed with no untoward results.

These observers noticed for the first time the production of waxes, which result from the esterification of the higher fatty acids with the higher alcohols formed as intermediate products in the oxidation. The complex reaction product contains, in addition to saturated fatty acids, quantities of hydroxy acids, and of unsaturated fatty acids formed from the latter by dehydration. The unsaponifiable residue contains only a little unchanged hydrocarbon, and consists chiefly of alcohols and ketones. In addition to taking extensive precautions with regard to analytical control and to reproduction of results in "repeat" experiments carried out under similar conditions, these observers have studied the oxidation of a pure hydrocarbon, namely, pentatriacontane, which is surely a move in the right direction.

*Problems awaiting solution.*—It is essential that a complete survey of the chemistry of the higher paraffins and olefines be made. Very definite information as to the occurrence of particular hydro-



carbons in different fractions of mineral oils is lacking, and must be obtained if we are successfully to oxidise them to fatty acids of definite composition and value. Methods of characterising the various mineral oils which are to serve as raw materials for this purpose must be available; and apparatus must be designed for obtaining any particular component or a definite mixture of components from natural oils on a practical scale.

With the chemistry of mineral oils established on a sound basis, it will be possible to turn with better prospects of success to the subject of the oxidation of a suitable fraction of a natural oil. More systematic attacks can then be made upon the problem of the influence of the various factors upon the oxidation, *e.g.*, the presence of a suitable catalyst, or of no catalyst at all, the presence of some alkaline agent to combine with and remove from the sphere of reaction the fatty acids produced, the effect of impurities, temperature, pressure, and so forth. Again, if known hydrocarbons can be isolated from mineral oils, experiments can be commenced upon the individual treatment required by the different classes of hydrocarbons. For instance, a hydrocarbon with about eighteen carbon atoms in its chain, but with an olefine linkage in the  $\alpha$ -position, would possibly require different oxidation treatment from that applied to a similar hydrocarbon in which the double linkage was in the middle of the chain, and, of course, in the latter case widely different products would result.

The study of the chlorination products of the saturated hydrocarbons should be made in greater detail even than has been done by Mabery (*loc. cit.*), and with the extremely difficult question of  $\alpha$ -chlorination settled, attention could be turned to the elimination of hydrochloric acid, and the formation of a double bond at the end of the hydrocarbon chain. Systematic research upon the problem of the oxidation of such a definite compound would then be possible, and would be much more satisfactory than investigations upon a complex mixture of unknown composition.

If, too, definite chloro-derivatives could be produced, one of the obstacles would be removed from the path of the Zelinsky process, although, in this process, the difficult problems of the starting, control and direction of the Grignard reaction, and the discovery of a suitable solvent would still remain.

These problems are, however, well in the future. Until the complicated mixtures of hydrocarbons of many different series which exist in the natural petroleum have been studied in great and almost wearisome detail, that is, until we know what we are working upon, and can separate different compounds which react specifically, we shall not solve the problem of the production of fatty acids, unless we do so by chance.

**Synthetic fats.**—Assuming that fatty acids could be produced in good yield, and sufficiently cheaply, by a process similar to one of those indicated above, the question of the formation of esters, glycerides or anhydrides suitable for edible purposes still remains. This should not be too difficult a matter. In the later years of the war 70–80 tons of "ester oil" per month was produced in Germany by the esterification of fatty acids (not synthetic) by means of ethyl alcohol or glycol.

Nevertheless, there are many problems of great importance connected with this question of synthetic fats. The various glycerides which constitute natural fats are many in number, and occur in a large variety of mixtures. To simulate a natural fat, with its mixtures of simple and mixed glycerides, would be no easy task, and the physiological study of compounded synthetic fats would have to be undertaken if such fats are to compete with natural fats in open market, and not merely to be supplied as prisoners' rations, or doled out to a starving population in blended margarine.

In the event of the fats obtained by the use of glycol or of mono- or poly-hydroxyl alcohols not being satisfactory, the esterification of synthetic fatty acids with glycerin would produce the analogues of natural glycerides. In this case, an opening would be provided for the development on an economic footing of the "Protol" glycerin process, in which suitable sugar-containing solutions are fermented under special conditions of alkalinity to produce glycerin, with alcohol, acetaldehyde, etc., as by-products.

Research would also be stimulated in the direction of using poly-hydroxyl alcohols, such as carbohydrates, for the esterification of synthetic fatty acids, and "fats" akin to the mannitan di-oleate, recently prepared by Lapworth, would result. Mannitan di-oleate was, of course, not produced by direct esterification, but there is no doubt that such an esterification is possible, and that the production of a fat of approved physiological value can be successfully accomplished.

**Natural fats.**—The problem of rendering petroleum available as a source of human energy by producing synthetic fats is therefore one which will only be solved after a very large amount of intensive and exhaustive research has been successfully undertaken. In passing, however, it is suggested that a more studied consideration of possible developments in natural vegetable-oil production would remove all fear of competition at the hands of the synthetic chemist. With careful selection of species, and systematic experiments in the production and application of suitable fertilisers, and with all the arts of the quickly-growing science of arboriculture, it should be possible greatly to increase the yield of oil per acre of ground cultivated. The fact that a similar study in the sphere of natural indigo has enabled the latter to compete with the synthetic product, so long its master economically, is significant in this connexion.

The production on a technical scale of fatty acids from petroleum hydrocarbons would appear to be possible, if remote; and were a successful process discovered, its general application would have but little effect upon the available supplies of mineral oil. Although the latter may be within measurable distance of exhaustion, the production of the million tons or so of edible oils and technically useful glycerides and fatty acids which are annually required in England would absorb such a small proportion of the hydrocarbon oil needed for other purposes that it would not become an important factor in petroleum economics. In August, 1920, the production of crude oil in America was about six million tons, and the consumption about seven million tons.

## THE EFFECT OF ALKALINITY ON THE USE OF HYPOCHLORITES.

E. K. RIDEAL AND U. R. EVANS.

It is, of course, well known that hypochlorites are used very largely for the following purposes:—(1) Bleaching; (2) sterilisation of drinking water; (3) treatment of water for "textile finishing;" to prevent growth of algae and consequent stains; (4) addition to water of swimming baths; and (5) chloride of lime is employed freely as a deodoriser for household use, for the cleansing of drains, and, to some extent, as a disinfectant.

In the first case (bleaching), where the rapid oxidation of colouring matter is desired, *e.g.*, for wood pulp, it is a common practice to allow the hypochlorite to act in presence of acid; the bleaching is

then very rapid. But in most other cases it is customary to apply small quantities of hypochlorite to the fluid to be treated, quite irrespective of whether the fluid be acid, neutral, or alkaline.

Now, in general, oxidising agents act most powerfully in the presence of acids. This is readily observed in the case of typical oxidising agents such as permanganates, dichromates, and perborates, which all tend to render the solution of the substance acted upon less acidic or more basic than it was before. In electro-chemical language this fact can be expressed by the statement that the oxidation potential of the agent is raised by an increase in the hydrogen-ion concentration.

The effect of acidity on the oxidising power of hypochlorite solutions is particularly marked, probably because, as has been suggested by one of us,\* hypochlorous acid is the effective oxidiser, the oxidising power of the salts and of the anion, (OCl), being but feeble.

Whether this explanation be accepted or not, the facts cannot be doubted. The matter is expressed quantitatively by the following data from the oxidation potential series:—

Potassium permanganate..	..	$E_H = +1.48$ volts.
Chlorine in acid solution ..	..	$E_H = +1.39$ "
Chromic acid ..	..	$E_H = +1.12$ "
Ferric chloride ..	..	$E_H = +0.96$ "
Chlorine in alkaline solution ..	..	$E_H = +0.86$ "

The table given indicates that chlorine in acid solution is nearly as powerful an oxidiser as potassium permanganate, and more powerful than chromic acid, whilst chlorine in alkaline solution (i.e., hypochlorites) is less powerful than ferric chloride, which is far from being a vigorous oxidising agent.

It is not generally known, however, that a comparatively small change in the hydron content of a water will produce a very marked change in the oxidising activity of the hypochlorite added. We have become very impressed with this fact in studying the dosage of different waters with hypochlorites by means of the electrical apparatus described by us some years ago.† We find that two waters, one an "acid" water containing free carbon dioxide and the other an "alkaline" water, such as occurs in the London basin, dosed with hypochlorite so that they contain the same amount of "available chlorine" (as measured by the ordinary iodometric method with titration in presence of acetic acid), give quite different results when allowed to flow through the electrical instrument. The acid water gives a large deflection, indicating high oxidising activity; the alkaline water gives a much smaller deflection, indicating that the oxidation potential has been depressed.

It is interesting to note that the dissociation constants of hypochlorous and carbonic acids and water have the following values:—

Hypochlorous acid ..	..	$K = 3.7 \times 10^{-8}$ at 17° C.
Carbonic acid ..	..	$K = 3.0 \times 10^{-7}$ at 18° C.
Water ..	..	$K = 0.7 \times 10^{-14}$ at 18° C.

Consequently, if sodium hypochlorite be added to water containing free carbonic acid, sodium carbonate and free hypochlorous acid will result; but in water in which the hydrogen-ion concentration is less than in hypochlorous acid, only a small amount of the oxidiser will be present in this active form.

Since the germicidal properties of the substance are in part due to its oxidising power, it seems likely that these also will be affected by the hydrogen-ion concentration of the water. It would be worth while, therefore, to consider whether, if

alkaline waters have to be treated with this class of steriliser, they should not at the same time be rendered just acid to litmus, or at least neutral, by the addition of nitre cake, aluminium sulphate, or even by treatment with carbon dioxide gas.

But there is another equally important point. Most hypochlorite liquors, whether made on the spot by treating bleaching powder with water, or brought from the factory as sodium hypochlorite solution, have an alkaline reaction. Just because they react less readily in alkaline solution, hypochlorites "keep" better in the presence of alkali, and the manufacturers are scarcely to be blamed if their products leave the works with more than a trace of alkali. But users should remember this fact, and neutralise the alkalinity of the solution before using it to treat contaminated water.

It is worthy of mention that some of the chloride of lime sold at small oil-shops and by grocers is largely decomposed before it reaches the user. Now the decomposition may proceed in various ways, but some at least of these cause the bleaching powder to become more alkaline as it decomposes. Supposing, therefore, that the bleaching powder has lost half its "available chlorine" (as measured iodometrically) by the time it reaches the consumer, it is not even true to say that the unfortunate user can make up for the loss in transit by employing a double quantity, since the extra alkali present has a depressing effect upon the oxidising properties of the substance remaining.

The variable character of the chloride of lime—as it reaches the consumer—appears to have very considerable public importance. One sample of chloride of lime (packed in paper), purchased by one of us at a local store, was found to contain only traces of available chlorine. Presumably the average user of the substance is not a chemist, and does not know that he is buying a valueless article. One cannot but conclude that, assuming the domestic use of chloride of lime by the customers of these stores to be necessary at all, they are running serious risks, since the safeguards which they imagine that they are taking are illusory. This is a matter to which public analysts would do well to pay serious attention. It is useless to blame the shopkeeper if manufacturers supply the substance in paper cartons permeable to carbon dioxide.

The strength of chloride of lime as it reaches the consumer in tropical countries has recently been discussed in this *Journal*.\* It is, however, remarkable how little the unstable character of chloride of lime—even at low temperatures—is appreciated. It decomposes very speedily if exposed to damp air or to carbon dioxide. On the other hand, sodium hypochlorite solution of a good brand has proved, in our experience, much more satisfactory as a source of hypochlorite so long as it is kept in a cool place, protected from light, and tightly sealed to exclude carbon dioxide.

In view, however, of the difficulty of preserving hypochlorites, it is worth considering whether liquid chlorine could not usefully be employed for many purposes where hypochlorites are now used. As long as it is in cylinder, chlorine cannot decompose, and when dissolved in water the main primary reaction is believed to be:  $\text{Cl}_2 + \text{H}_2\text{O} = \text{HCl} + \text{HClO}$ . Both of the substances produced are acid, and one is a strong oxidising agent.

**Summary.**—The oxidising power of hypochlorite solutions is greatly depressed by the presence of alkali. This fact should be borne in mind when it is proposed to use hypochlorites for treating alkaline waters; and the simultaneous addition of nitre cake is suggested.

Where alkali has been added as a stabiliser to commercial hypochlorite solutions, it may be advis-

\* R. K. Eideall, *J. Chem. Tech.* (1912), 141.

† As given in W. Ostwald's "Outlines of General Chemistry."

‡ R. K. Eideall and U. E. Evans, *Analyst*, 38 (1913), 353.

\* J. Rettle, J. L. Smith, and J. Ritchie, *J. Soc. Chem. Ind.*, 37 (1918), 2117.

able to neutralise before using the liquor for dosage. The instability of chloride of lime, and the poor quality of many of the samples supplied in paper packages by small stores, are matters of importance.

## MEETINGS OF OTHER SOCIETIES.

### ROYAL PHOTOGRAPHIC SOCIETY.

At the meeting held on February 8, arranged by the Scientific and Technical Group, Mr. T. Thorne Baker read a paper, by himself and Dr. M. Levy, on "High-Speed Radiography." A short statement on the present possibilities and requirements in X-ray work, with special reference to the use of calcium tungstate intensifying screens, led to a description of the new rapid X-ray plate. This is a double-coated plate, the lower film being light-sensitive, the upper one containing fluorescent calcium tungstate. As it is necessary to remove the top film before development, the lower film is specially hardened to stand prolonged soaking at 110° F., and a soft gelatin, dissolving easily in water at 95° to 105° F., is used for the upper film. The calcium tungstate is fired at a special temperature to obtain maximum fluorescence. The grains are sifted and only those between certain limits of size are used; smaller grains give less intense fluorescence and larger grains tend to make the image granular. The coating of the upper film is so arranged that the calcium tungstate settles to the bottom of film into close contact with the emulsion surface; this ensures maximum effect of the light emitted and enables the use of a much smaller quantity of calcium tungstate than is needed for an intensifying screen. The reduction of exposure required by this plate in comparison with an ordinary X-ray plate depends upon several factors and varies from 1/10" to 1/25"; better results are obtained with a hard tube than with a soft one. It was suggested that the new plate would be particularly useful in radio-metallurgy.

Mr. F. F. Renwick gave a short abstract of a paper received from Mr. Raymond Davis, of the U.S. Bureau of Standards. The multiplying factors of photographic light-filters are obtained by an apparatus comprising a movable lamp and a series of mirrors permitting the illumination of two adjacent portions of a photographic plate by lights of known relative intensities. A series of exposures is made with the filter in one path, the results plotted, and the position of equality determined from the curve obtained.

Mr. F. C. Toy, of the British Photographic Research Association, described a monochromatic illuminator designed chiefly for use with a mercury arc lamp. This is a simple device enabling the separation of any two bands of a spectrum, and the variation of the lengths of the two bands used can be adjusted to equal intensities by the aid of a thermopile and galvanometer. Known variation in relative luminosities of the two beams is then obtained by varying the distances of the recombining lens and the plate which receives the recombined image.

### THE PHYSICAL SOCIETY.

The annual general meeting was held at the Imperial College on February 11, and after reports had been presented and officers elected, etc., a discussion was held on "Absolute measurements of electrical resistance and instruments based on the temperature-variation of resistance."

Sir Richard Glazebrook traced the historical development of absolute measurements of electric resistance, and Prof. Callendar dealt with the com-

pensated resistance bridge and instruments for the measurement of radiation. Among the thirteen other papers read, mention may be made of the following as having some application in chemical industry. These papers illustrated the diversity of ways in which one of the fundamental devices of electrical measurement—the Wheatstone bridge—has recently been applied in the measurement of various physical quantities.

A liquid depth-gauge, enabling a reading of the depth of liquid in a vessel to be made at any distance from the vessel, was described by Mr. E. A. Griffiths. The instrument consists essentially of a Wheatstone network of four conductors, two of which, in opposite arms of the bridge, are wires of the same material, extending to the depth of the vessel. One of the wires is completely surrounded by a sheath to protect it from contact with the liquid. The other is similarly surrounded, but the sheath is provided with holes near the bottom and top, so that the wire is in contact with the liquid over a length equal to the depth of liquid in the vessel. The wires are heated by the current in the bridge and the original balanced condition of the bridge when the vessel is empty, is disturbed to an extent dependent upon the depth to which the one wire is in contact with the liquid. Constancy of current in the bridge is maintained by the use of a ballast resistance of iron wires enclosed in an evacuated space. The galvanometer employed is graduated so that its deflections indicate directly the depth of liquid in the vessel. The whole device is made up in a compact form, the indications are independent of temperature, and can be transmitted to any distance.

A strong and portable form of hot-wire anemometer was exhibited and described by Mr. J. T. McG. Morris. The Wheatstone bridge employed is composed of four nickel wires, alternate arms being shielded. When the device is exposed to an air stream the initial balanced condition of the bridge is upset owing to the cooling effect of the stream on the exposed wires, and the galvanometer deflections, after suitable calibration, indicate the velocity of the air stream in which the instrument is immersed. This device has been used to investigate the ventilation of mines, and should be applicable wherever measurements of air velocity are required (e.g., air supply to producers, power plant, etc.), its indications being much more easily read than those of the Pitot tube. A form of hot-wire anemometer enabling the velocity of a slow-moving stream of gas to be determined, both as regards magnitude and direction of flow, was exhibited and described by Dr. J. S. G. Thomas (cf. J., 1920, 109 a). A combined form of instrument of the anemometer type, in which the exposed wire functioned first as a thermometer and then as the exposed wire of a hot-wire anemometer was described by Mr. A. H. Davis. The indications of such a device enable the heat loss by convection from heated bodies to be accurately determined. It has been used in investigating the thermal efficiency of various systems of cold storage.

Dr. G. A. Shakspear exhibited and described his gas permeameter, which contains a bridge-device of four fine wires similar to that mentioned above. Owing to the varying thermal conductivities of different gases, the heated condition of appropriate wires is dependent upon the composition of the gas in which they are immersed. After suitable calibration of the instrument, the galvanometer deflections indicate the percentage amount of any one constituent of the mixture present. Hydrogen is pre-eminent among gases for its extremely high thermal conductivity (hence the cold sensation experienced on plunging the hand into the gas), and the author's device has been extensively used to determine the purity of the hydrogen supplied for aeronautical purposes in the investigation of the

permeability of balloon fabrics, etc. A modification of the permeameter, in which the indicating instrument is suitably calibrated, constitutes the CO<sub>2</sub>-recorder described and illustrated by Dr. H. A. Daynes. Prof. L. Hill exhibited the "calcometer," a bridge device enabling a record to be obtained of the state of the atmosphere as affording comfort or otherwise.

The familiar hair type of hygrometer has been modified by Dr. E. Griffiths so that its indications may be transmitted to a distance. The length of the hair employed is dependent upon the hygrometric state of the atmosphere, and the changes of length accompanying change of hygrometric state are transmitted mechanically to a pointer which makes contact with a long length of wire, the point of contact varying with the length of the hair. The two portions of the wire, one on either side of the point of contact, constitute two adjacent arms of a Wheatstone bridge, and it is evident that the indications of the galvanometer device employed will serve, after suitable calibration, to indicate the hygrometric state of the atmosphere surrounding the hair.

#### THE CHEMICAL SOCIETY.

At the ordinary scientific meeting held on February 17, ninety-seven new Fellows were elected, and Sir J. J. Dobbie, retiring president, made the following announcements:—

The Longstaffe Medal has been awarded to Prof. J. F. Thorpe. On the evening of the Annual General Meeting, March 17, the Anniversary Dinner will be held at the Hotel Cecil, Strand, W.C., at 7 p.m., to which the past-presidents who have attained their jubilee as Fellows of the Society will be invited as guests of honour. Applications for tickets (14s., exclusive of wine) should be made to the Assistant Secretary, at Burlington House, by March 10, and the presence of ladies as guests will be welcomed. The Council has decided to publish the list of physico-chemical symbols drawn up by the International Association of Chemical Societies.

Mr. L. J. Hudleston and Prof. H. Bassett contributed a paper on "Equilibria of Hydrofluosilicic and Hydrofluoric Acids," which contained a record of experiments on the titration of these acids with alkali in presence of phenolphthalein or methyl orange, and a theoretical interpretation thereof. Mr. Hudleston stated that they had not been able to purchase even approximately pure hydrofluoric acid, the samples supplied containing over 50 per cent. of hydrofluosilicic acid. Further, the "A.R." test for determining silica in solutions of hydrofluoric acid was found to be valueless.

Mr. W. C. Reynolds summarised briefly two papers on Interfacial Tension, in the first of which he described a capillarity method of measuring it in C.G.S. units, and adduced results comparing it with the dynamical or "drnp" method. In the second he showed that the interfacial tension between two pure liquids, A and B, is the difference between the surface tension of A saturated with B and the surface tension of B saturated with A; that the interfacial tension between pure liquids is constant at any given temperature; and that aqueous solutions of crystalloids behave like a pure liquid. The interfacial tension between aqueous sols of soap and colloids in contact with benzene was found to vary with the age of the contact surfaces. They probably conform to the law at the moment of contact, but owing to surface concentration the tension changes. In soap sols the concentration is complete in a few minutes, but in colloidal sols generally it proceeds for several hours. In cases where the interfacial tension is not constant, its measurement affords a means of detecting chemical or electrical action, or the presence of impurities in liquids.

Mr. F. W. Atack criticised the Hantzsch-Werner hypothesis of the geometrical isomerism of carbon-nitrogen compounds, and dealt with the constitution of oximes, and Prof. Bassett communicated a short note on the system: picric acid—phenylacridine.

#### THE FARADAY SOCIETY.

A joint meeting of the Faraday Society and the Manchester Literary and Philosophical Society was held in Manchester on February 11. The chair was first taken by Sir Henry A. Miers, and then by Prof. A. W. Porter.

Dr. Allan Ferguson read a paper entitled "Studies in Capillarity. I. Some General Considerations and a Discussion of Methods for the Measurement of Interfacial Tensions." He pointed out the importance of accurate measurements of surface tension in view of the developments in colloid chemistry, and stated that a critical study of the various methods in systematic use had shown the following to be the most promising:—(1) The measurement of large drops and bubbles. (2) The measurement of the maximum pressure required to release a bubble of air from the end of a capillary tube immersed in the liquid. (3) The measurement of the maximum pull on an anchor ring, which is immersed in the liquid and then slowly withdrawn from it. The capillary-tube method was discussed, and it was shown that a comparatively simple mathematical analysis for the ascent of a liquid in a capillary tube gave the sufficiently accurate expression:—

$$2a^2 = rh \left( 1 + \frac{1}{3} \frac{r}{h} - 0.1258 \frac{r^2}{h^2} \right),$$

where  $a^2$  is the specific cohesion, and  $h$  the height to which the liquid rises in a tube of radius  $r$ .

Dr. Ferguson then read a second paper by himself and Mr. P. E. Dowson on "A Modification of the Capillary-tube Method for the Measurement of Surface Tensions." In this new method the meniscus is forced down to the end of the capillary immersed vertically in the liquid, and the pressure required to effect this is measured on a manometer. This procedure avoids most of the difficulties of the usual method. Temperature measurements and control become simple, and calibration of the capillary is not necessary, for measurements are always made in one definite position—at the end of the tube. Any manometer of sufficient delicacy may be used, such as a differential manometer, or one of the simple sloping-tube type. The following values were obtained for the surface tension in dynes/cm.:—Benzene (15° C.) 29.66; toluene (15° C.) 29.23; methyl propionate (15.5° C.) 26.09.

In the long discussion which followed, Prof. Porter said he had had considerable experience in measuring the surface tension of mercury; he had photographed large drops and had found that the method of illumination was an important factor. He regarded the new method given by Dr. Ferguson as much more satisfactory than that of measuring the capillary rise. Mr. F. C. Thompson said that he was especially interested in the applications of surface tension to metallurgical problems and gave examples in connexion with the constitution of steel. Dr. R. S. Willows said that surface tension data were of particular importance in connexion with the formation of emulsions, and he asked for a simple method he could apply. Prof. Haldane Gee referred to the work of Quincke, who is still working at the age of 87 on the subject at Heidelberg, and who has recently advanced a new theory of the constitution of glass depending on surface tension. Prof. A. Lapworth referred to the values obtained by Dr. Ferguson and Mr. Dowson for benzene, which were found to change with time; this required further investigation.

## ASSOCIATION OF BRITISH CHEMICAL MANUFACTURERS.

### LORD MOULTON ON "KEY" INDUSTRIES.

A luncheon was given by the Association of British Chemical Manufacturers on February 16, at Prince's Hotel, W., in honour of its president, the Rt. Hon. Lord Moulton, for his services to chemical industry during and after the war. Among those present were the Rt. Hon. Sir Alfred Mond, the Rt. Hon. Lord Inverforth, Sir William Pearce, Sir William Pope, Sir Henry Birchenough, Sir Sydney Chapman, Sir Philip Magnus, Sir H. McGowan, Prof. W. H. Perkin, Prof. H. E. Armstrong, Dr. M. O. Forster, Mr. Max Muspratt, and Mr. W. J. U. Woolcock.

Sir William Pearce, who presided, in proposing the health of Lord Moulton, referred to his accomplishments as senior wrangler, great lawyer, administrator, chemist and scientist, and said that the recent passage of the Dye-stuffs (Import Regulation) Bill through the House of Lords was largely due to his advocacy. That Act must now be supplemented by further legislation to secure the future of the synthetic organic chemical industry, a branch of the main industry which had long been recognised and fostered by the German Government as one of the most important. It was essential that our Government and country should recognise its supreme value and legislate accordingly.

Lord Moulton, in his reply, said that this was a most critical moment in the existence of the British chemical industry and, through it, in the existence of England. During four years of the war he had endured terror of what the German chemical industry had enabled our enemies to do; he was always urging the Treasury to buy ahead from Chile the nitrate which the Germans were making in unlimited quantities at home, and he was afraid to think what would have happened if Germany had concentrated her submarine attack on that narrow line through the Panama Canal by which we obtained our supplies. In this, and in many other ways, we were reminded of the consequences of the neglect by the British nation and the British Government of the chemical industries during the 30 or 40 years before the war.

Referring to the old war-cry of Free Trade v. Protection, Lord Moulton said that we in this country were too fond of adopting political formulae and calling them principles. As an old Free Trader, he used to say that the quickest way to obtain national wealth and to enjoy it was to buy in the cheapest market. That definition must now be elaborated as follows:—"If your main object is the rapid acquisition of wealth and the enjoyment of wealth, and if you are sure of continuous peace, then the best way is to buy in the cheapest market." Unfortunately we could not rely on a continuous peace, and the rapid acquisition and enjoyment of wealth could only be regarded as the highest good if it included the perfect development of our greatest asset—our own people. We had allowed ourselves to grow up as a nation utterly insufficient for ourselves, depending on others for the supply not only of manufactured goods, but also of many raw materials. When he looked back on the confident utterances of the old-time Free Traders, he was surprised that some of them did not insist that we should contract with Krupp for our war material and accept his tender if it were the lowest. Now they knew that they dare not be dependent on a foreign country for munitions of war; but they did not recognise that there were other things just as essential for use in peace. We no more dare leave our great industries at the mercy of a foreign

country than we dare trust to a foreign country for our guns and ammunition. It was quite impossible to prop up every industry that did not make itself industrially remunerative, but when as a result of long national neglect the country had called on industries to initiate and develop new branches during the war, to expend capital on them and to develop research, it was the country's duty to protect those industries until the crisis was passed; and to think that they could withstand unrestricted competition while yet in a state of infancy was sheer folly. The problem would not be permanently solved by means of subsidies or import duties. His experience during the war had given him unlimited faith in British chemistry, both theoretical and industrial, and he declined to regard our chemists as in any way inferior, except as regards experience, to those of any other nation. He had always supported the system of licensing under which protection was secured for British goods so long as they were produced at home at fair prices and of equal quality; such a system would afford our manufacturers a breathing space in which to develop, not as a favour but as a right, their power of production until they needed no help and feared no enemy.

## CORRESPONDENCE.

### THE FINE CHEMICAL INDUSTRY.

(Communicated by the Association of British Chemical Manufacturers.)

SIR.—We, the undersigned, makers of fine chemicals, extended our research and manufacturing plant at the urgent appeal of the State during the tremendous years that followed 1914, in order to secure those essential products of science without which victory could not be attained. The full record of wartime achievements in the sphere of fine chemicals would read like a romance were the full history disclosed.

This key industry is now endangered, and unemployment is rife in it, owing to the flood of imports from abroad which, among other circumstances, the present condition of the exchanges makes devastatingly possible.

We desire to submit to public knowledge the following facts:—

1. The application of chemical science to industry is a necessary factor for the preservation of our world position in commerce. It is also true to say, not only that the chemist and the laboratory are in this country of a fine chemical industry not inferior pivotal in the development of our industrial and commercial resources and wealth.

2. At very considerable cost, essential plant was erected during the war-years, and all of it can now be put to peaceful purposes for the continuance in this country of a fine chemical industry not inferior to Germany's. But, under the present unfair economic circumstances, this plant must become idle and useless, unless the State takes measures for its safeguarding.

3. The fine chemical industry, mobilised and expanded in the hour of the nation's needs, provided during the war—to give a few examples—(a) essential medicinal preparations for naval, military, and civilian hospitals; (b) the higher forms of explosives and gases; (c) photographic chemicals for aerial use; (d) research chemicals, without which the development of many new industrial processes would have been impossible.

4. British science is no whit inferior to that of competing nations, nor are British chemical manufactures inferior in quality.

We feel it is our bounden public duty to make known to the country the national importance of an industry which is indeed a key to the unlocking of many doors of economic wealth in the future.—We are, etc.,

STAFFORD ALLEN & SONS, LTD.  
A. BOAKE, ROBERTS & CO., LTD.  
BOOTS PURE DRUG CO., LTD.  
THE BRITISH CELLULOSE & CHEMICAL MANUFACTURING CO., LTD.  
THE BRITISH CYANIDES CO., LTD.  
THE BRITISH DRUG HOUSES, LTD.  
BRITISH VEGETABLE PRODUCTS, LTD.  
BURROUGHS, WELLCOME & CO.  
W. J. BUSH & CO., LTD.  
J. M. COLLETT & CO., LTD.  
EVANS, SONS, LESCHER & WEAR, LTD.  
GRAYS DYES AND CHEMICAL WORKS.  
HOPKIN & WILLIAMS, LTD.  
HOWARDS & SONS, LTD.  
JOHNSON & SONS, MANUFACTURING CHEMISTS, LTD.  
LONDON CHEMICAL WORKS, LTD.  
MAY & BAKER, LTD.  
THOS. MORSON & SON, LTD.  
PIERSON, MORRELL & CO., LTD.

Feb. 19, 1921.

## NEWS AND NOTES.

**Petroleum Research.**—The American Petroleum Institute intends to create a department of research, and its board of directors has recommended that three engineers be appointed, one to investigate oil-field problems, another to investigate the refinery problems, and a third to deal with problems relating to the distillation of petroleum and its products.

**Colloid Chemistry.**—Attention is drawn to a discussion of research problems in colloid chemistry by Dr. W. D. Bancroft which began in the January number of the *Journal of Industrial and Engineering Chemistry*. In his first communication Dr. Bancroft discusses seventeen problems under the general head of "Adsorption of Gas or Vapor by Solid," fourteen under "Contact Catalysis," three under "Adsorption of Vapor by Liquid," and nine under "Adsorption of Liquid by Solid."

**Improved Yield of Methyl Alcohol in Wood Distillation.**—A report will soon be published by one of the Government laboratories outlining experiments which have indicated the possibility of practically doubling the yield of methyl alcohol from the distillation of certain woods if a very small percentage of a certain chemical compound be first sprayed upon the wood. Encouraging results have been obtained in the laboratories from the use of sawdust, but it remains to be seen whether any practical difficulties will limit the size of pieces which can be treated. If such improved yields can be obtained commercially in the treatment of wood waste, certain perfected methods for the distillation of these wastes will be put on a very sound basis.

**Ethyl Alcohol from Wood Waste.**—The United States Forest Products Laboratory at Madison, Wisconsin, has prepared a pamphlet describing the production of ethyl alcohol from wood waste. The pamphlet includes estimates of plant requirements and cost data compiled from recent experiments at the laboratory. It is shown that with 180 tons of wood waste available daily, 3600 galls. of alcohol can be produced at a cost approximating 25 cents a gallon. The soft woods give the highest yields of 95 per cent. alcohol per ton of wood; white spruce

heads the list with 25.8 galls., longleaf pine is next with 25.1 galls. The smallest yields are obtained from hard maple, red oak, cottonwood, and slippery elm, the first of these yielding but 9.1 and the last 6.0 galls. per ton.

## AUSTRALIA.

### South Australia.

**Lignite Deposits.**—A recent report of the Department of Mines states that the rise in the cost of imported fuel during recent years and the dislocation of industry through the interruptions in marine transport have drawn attention to all domestic sources of supply. In this connexion there is a revival of interest in the lignite deposits near Moorlands, where prospecting is now being actively pursued.

The lignite exposed in the workings has a somewhat variable appearance. The dull earthy and structureless body contains fragments of partly carbonised wood in which the fibres are plainly visible. There are a few irregularly shaped pieces of lustrous lignite and many traces of resin throughout the bed. The coaly matter contains much pyrites disseminated throughout the body of the carbonised wood. The following methods of utilising this lignite have been suggested:—(1) The generation of power with the aid of steam produced from specially designed furnaces burning the raw lignite. (2) The generation of producer gas from the raw fuel and the recovery of by-products. (3) Utilisation of the pulverised fuel for raising steam. This method involves the artificial drying of the lignite. (4) In the form of briquettes manufactured from the lignite after drying. (5) The carbonisation of the lignite with recovery of the by-products by distillation.

In view of the vital importance of fuel supplies to the State, the Government has purchased a modern drill and boring operations, designed to prove the extent and quality of the brown coal deposits of the State, are in progress in the vicinity of Adelaide. The Government has also allocated £5000 for experiments on pulverised coal from Leigh Creek. The tests will be designed primarily to ascertain the value of this coal for use in railway locomotives, but they will be extended to other domestic fuels, such as the lignite of Moorlands.

## BRITISH INDIA.

**First Forecast of Winter Oil-seeds.**—The total area under rapo and mustard, excluding the mixed crop in the United Provinces, is estimated at 2,835,000 acres, or 13 per cent. below the estimated area in 1919-20. The percentage distribution of this area is as follows:—United Provinces 40.2, Bengal 19.4, Punjab 16.2, Bihar and Orissa 11.7, Assam 4.6, Bombay and Sind 4.7, North-West Frontier Province 1.8 per cent. The total estimated area under linseed is 1,755,000 acres (excluding the mixed crop in the United Provinces), representing a decrease of 23 per cent. The proportion sown by each province is:—Central Provinces and Bihar 35.3, United Provinces 29, Bihar and Orissa 23, Bengal 5.5, Bombay 5 and Punjab 1.2 per cent. Owing to deficient rainfall, the conditions for sowing were generally unfavourable. Rain is urgently wanted in most of the provinces and the condition and prospects of the crops are only fair.—(*Indian Trade J.*, Jan. 7.)

## JAPAN.

**Petroleum Production.**—The output of crude petroleum in Japan during the first six months of 1920 was 38,181,675 galls., compared with 38,891,918 galls. during the same period of 1919. Over 99 per cent. of the production was obtained in the prefectures of Niigata (Echigo) and Akita.—(*Sekiyū-Jihō*, Nov., 1920.)



## CANADA.

**Maple Sugar "Sand."**—During the evaporation of maple sap to the state of syrup, a precipitate technically known as "sand" is deposited. It contains 60–80 per cent. of calcium malate, and its annual production from all the trees in the United States and Canada is estimated to be 100–330 tons, corresponding to 50–165 tons of calcium malate. Previous to the war it was bought by a German manufacturer of fine chemicals, and efforts are now being made to interest chemists in other countries in the possible uses to which this by-product may be put. As the acid constituent of baking powder, calcium bimalate gives excellent results, but it can hardly be produced as cheaply as calcium acid phosphate or even cream of tartar. Other applications, either of the normal or acid salt or of malic acid, are being investigated. In the case of no other source of malic acid can extraction be so readily effected, it being necessary only to convert the normal to the acid salt, and the latter being much more soluble in hot than in cold water is easily separated in a comparatively high state of purity by re-crystallisation. (*Proc. 27th Annual Meeting of the Vermont Maple Sugar Makers' Association.*)

## FRANCE.

**Industrial Notes.**—*Chemical Industry.*—The closing down, either partial or complete, of factories in the dyeing, bleaching, and leather industries has led to a weaker demand which has affected prices. Wholesale dealers, fearing a sudden fall in prices, are eager to dispose of their stocks, even on unfavourable terms. On the whole, however, there is no panic and prices are steadily finding their level without any State interference. German competition is proving a great stimulant and many firms are extending their plant in order to increase output. The Tencé firm, which has a plant at Aubervilliers, is building a new factory at Bernes, near Persan-Beaumont, for the production of sulphates, chlorides, nitrates, and of hydrochloric, sulphuric and nitric acids in both pure and commercial grades. The Compagnie Nationale des Matières Colorantes has just begun to produce alizarin and other basic dyes, in addition to indigo, at its works at Villers-Saint-Paul.

*Metallurgy.*—The outlook in the metallurgical market remains practically unchanged; iron ore is in poor demand and there are large stocks of minette. The warning given to German firms that unless they supply France with the coal and coke she needs their supplies of iron ores will be cut off, have caused them to seek for new sources of supply, notably in Sweden. Lorraine is now supplying the Ruhr district with only 10 per cent. of its requirements, which compares with 50 per cent. before the war. On the whole, there is a feeling of confidence in French metallurgical circles. Owing to stocks of raw material being very low, the French will be able to take advantage of the fall in prices in replenishing them. Prices of wrought iron and steel have fallen considerably, and French pig iron is now sold at a lower price than the British. The chief competition comes from Germany, where production costs are lower than in France.

## GENERAL.

**Research Chemicals and the Dyestuffs Act.**—In response to an inquiry made by the Institute of Chemistry, the Board of Trade has intimated that it will issue general licences under the Act to approved research institutions for the importation of small quantities of organic intermediate products. The licences will cover periods of three months and be limited only as to total quantities. A detailed return of the quantities of each product actually imported must be made at the end of the three-monthly period.

**The Textile Institute.**—Colonel F. R. McConnell, of Manchester, is to succeed the late Sir A. Herbert Dixon, Bart., as president of the Institute. At the invitation of the Basle Chamber of Commerce the Annual Spring Conference will be held from May 17 to 20 at Basle, in co-operation with several leading textile organisations in Switzerland. During the conference lectures will be given on "The Dye Industry and its Future," "The Industrial Application of Electricity," "The Financial Situation," and "The Structure of Textile Fabrics." The programme includes facilities for visits to textile and other works.

**Industrial Alcohol in Switzerland.**—At a meeting held on January 5, convened by the Swiss Society of Chemical Industry and attended by representatives of all the larger firms which use alcohol in their manufactures, a resolution was passed condemning the decision of the National Assembly to extend the alcohol monopoly to industrial spirit. The meeting protested that the decision had been made without consultation with those whom it would affect, and affirmed that at the present time the restrictions attending such an extension of the monopoly would be fatal to the country's export trade. (*Schweiz. Chem.-Z., Jan. 25, 1921.*)

**Patents in Greece.**—The first Greek patent law, passed early in the autumn of 1920, provides that every patent shall be granted on priority of application, shall cover only one invention, and have a life of 15 years, reckoned from the day following the date of application. The ownership of a discovery made in an industrial or commercial establishment is vested in the discoverer, and patents must be satisfactorily exploited within three years after coming into force, although this does not apply to nationals of countries that offer reciprocal treatment. In the event of a patent being declared of public utility, the owner must allow others to exploit it on payment of compensation. Foreign applications must be made through an agent resident in Athens and be subject to local jurisdiction. Where patents are registered in countries that allow reciprocal treatment the Greek law allows 12 months' priority from the date of the patent. Applications must be in Greek and be accompanied by an official certificate from the country in which the patent is registered. Patent specifications will be published at once, but may be kept secret for a year if requested. (*Z. angew. Chem., Dec. 17, 1920.*)

**State of the Swedish Chemical Industry at the end of 1920.**—The situation in the Swedish chemical industry has been growing worse, and in consequence of falling prices in the world's markets and of the high wages now paid in Sweden, manufacturers have either to close down or to produce at a loss. Many branches of the industry are demanding increases in import duties. Hitherto ammonium sulphate has been imported freely, but the body which represents all the Swedish manufacturers of this fertiliser is asking for an import tax of 15 kronor per quintal (about £8 10s. per long ton, at par); the yearly output of ammonium sulphate, about 9000 metric tons, is far in excess of the home demand, as cyanamide is chiefly used for nitrogenous manuring, and the export trade is suffering severely from German competition. No improvement has taken place in the soap industry; sales of soft soap are poor and prices have fallen; most of the factories hold big stocks of soap and produce only sufficient to keep plants in operation. The amalgamation of the Kväveindustrie (nitrogen industry) and the Trollhätte Cyanidverk companies has fallen flat, and the necessary new capital not having been obtained, the directors have notified the suspension of operations. The Swedish Government has appointed an expert advisory commission to investigate the bad economic situation. (*Z. angew. Chem., Jan. 7, 1921.*)



"Monazite (1913-1919)" (pp. 15, price 6d.) has been recently published by the Imperial Mineral Resources Bureau as an advance "part" of its annual volume on the Mineral Resources of the British Empire and Foreign Countries. Monazite, essentially a phosphate of the cerium earths, owes its commercial value to the presence of a variable amount of thorium which is extracted by chemical treatment and marketed in the form of thorium nitrate for use in the manufacture of gas mantles. The mineral is widely distributed in nature, but few deposits have been discovered which contain thorium in sufficient quantity to justify commercial exploitation. It is found in the form of an alluvial and associated with other minerals, such as magnetite, ilmenite, garnet, and zircon, from which it is separated by hydraulic and electromagnetic methods to produce the concentrate as it is marketed. With the exception of small quantities of thorite and thorianite obtained from Norway and Sweden, the first deposits to be worked for monazite were those situated in the Carolinas. These deposits, patchy in distribution and poor in quality, could not compete with those discovered later in Brazil, which supplied the world's requirements until the monazite from Travancore, India, appeared on the market about the year 1911. The earlier history of the Travancore deposits, and the final obliteration of the German interests, have been already fully referred to in the pages of this *Journal* (cf. S. J. Johnstone, J. 33, 55, 35, 811, and 37, 373 n). The separated monazite is the highest grade on the market, containing about 8½ per cent. of thorium as compared with 6 per cent. in the best of the concentrates obtained from the Brazilian sands. To illustrate the variable thorium content, reference may be made to monazite discovered in such widely separated countries as Nigeria, Queensland, Malaya, and the United States, which, even when concentrated by the most perfect means commercially available, commonly contain no more than 4 or 6 per cent., and frequently considerably less. The present price of monazite is stated to be £4 12s. 6d. per unit of thorium per ton at the mine in Travancore. The c.i.f. value at European ports depends upon the cost of freight. In June, 1920, it was about £6 10s. per ton.

Statistics quoted include the world's production of monazite, imports into the United Kingdom (for some unstated reason these are given in values), and into the United States.

#### World's Production of Monazite.

	1913.	1914.	1915.	1916.	1917.	1918.	1919.
India	1235	1186	1108	1292	1940	2117	2000*
Ceylon	—	—	—	—	—	20	—
Brazil	1415	590	433	—	1118	491	—
United States	—	—	16	17	—	—	—

\* Estimated.

Thorium nitrate, worth about 23s. per kg. before the war, is now selling at about 38s. Before the war this country was dependent upon Germany; since the war and up to within the last year or two nitrate was imported from the States, but now British manufacturers apparently supply home requirements.

#### Value of Imports and Exports of Gas Mantles to and from the United Kingdom.

Year.	Imports.	Exports.	
		Domestic produce.	Foreign produce.
1913	302,576	32,566	5050
1914	199,299	47,400	4359
1915	228,075	98,960	4848
1916	57,067	144,292	7491
1917	24,106	94,058	—
1918	26,815	103,297	—
1919	81,771	88,893	—

The mixture of oxides in gas mantles is said to be derived from a mixture containing about 99 per cent. thorium nitrate and 1 per cent. cerium

nitrate. Large quantities of cerium compounds are obtained as by-products in the manufacture of thorium nitrate, and new uses for this product, hitherto largely wasted, would be welcomed by the industry. At the present time the cerium is used for the manufacture of the alloy ferro-cerium, which usually contains about 30 per cent. iron, and which is used in the manufacture of sparking devices for carbide lamps, gas jets, and automatic cigar lighters. Cerium has also been suggested as a deoxidiser in the manufacture of steel and also in the "thermit" process for the reduction of metallic oxides, and its salts are used in the manufacture of flaming electric-arc lamps, in glass making, ceramics, and in medicine. Monazite is the chief source of mesothorium, which closely resembles radium in its properties. The amount present is only a few milligrams per ton of the monazite and apparently it is not extracted by the English manufacturers of thorium nitrate, although at least two manufacturers are producing mesothorium from monazite in the United States. Mesothorium is sold on the basis of its radio-activity compared with radium bromide, and was worth (June, 1920) from £6 to £8 per milligram.

In this connexion reference may be made to three recent publications in the United States, viz., "The Incandescent Gas-Mantle Industry" (United States Tariff Commission, 1920), "Thorium, Zirconium and Rare-Earth Minerals in 1919 (U.S. Geological Survey, 1920), and "The Mineral Industry" for 1919 (New York, 1920). The last states that during the war American manufacturers of thorium nitrate firmly established themselves, and every effort is now being made to pass protective legislation to prevent the flooding of the American market with low-priced German thorium nitrate, and claims that it is evident that a high duty should be placed on all imported thorium nitrate if the welfare of the American industry is to be considered, as the German producers have the advantages of cheap labour and longer hours, and the American producers cannot hope to compete with them. In the States prices remained comparatively firm throughout 1919, monazite sand ranging from \$38 to \$42 per unit, duty paid, equal to \$228 to \$252 per metric ton for a sand carrying a minimum of 6 per cent. of thorium. The prices of the nitrate remained steady throughout the year, ranging from \$3.75 to \$4 per lb. A small quantity of ferro-cerium, estimated not to exceed 10 tons per annum, is produced in the United States.

According to "The Incandescent Gas Mantle Industry," the productive capacity of the American plants shows an increase of 300 per cent.; 149,850 lb. of thorium nitrate was produced in 1913, and 439,304 lb. in 1917. The imports amounted to 1880 lb. in the latter year and 112,000 lb. in 1913. The same publication remarks that an *ad valorem* duty has been levied on the crude raw material, the refined intermediate product and the finished article, and if the duty is calculated on the basis of the content of the constituent of principal value, namely thorium, specific rates of duty obtaining in 1914 were as follows:— Monazite sand, \$0.315 per lb. of thorium contained; thorium nitrate, \$1.44 per lb.; and gas mantles, \$7.48 per lb.

**Finland in 1919.**—The chief natural resources of Finland is timber, which covers an area of 77,200 square miles and is estimated to last about 145 years on the basis of an annual production of 300,000 tons of chemical pulp and 200,000 tons of mechanical pulp. Water power is also abundant, but only 150,000 h.p. is in use, about 1,460,000 h.p. remaining unutilised. Several mines have been worked in East Finland, including the Outokumpu copper pyrites mine, which is estimated to contain at least 230,000 tons of copper and over 2 million tons of combined sulphur (cf. J., 1919, 27 B, 47 B).

Wood pulp and paper constitute the most important industries. Trade in these commodities was formerly mostly with Russia, although since 1903 there has been a steady market for Finnish wood pulp in Denmark, Germany, Holland, Belgium, England, and France. The following table gives the quantities (long tons) of mechanical pulp and board exported in 1913 and 1919:—

Wood Pulp.	1913.	1919.
Wet .. ..	8,916.5	12,504
Dry .. ..	39,750	50,861
Total .. ..	48,666.5	63,365
Pasteboard (wood) ..	52,884	910

Nearly all the wood pulp and board exported is supplied by the twenty-three mills belonging to the Finnish Wood Pulp Mill Union (Finska Trasliperi-föreningen), which has a combined capital of 100 million Finnish marks (about £4,000,000 at par), and a total annual capacity of about 150,000 tons. Of the 1919 production, 5518 t. was sold in Finland and 74,609 t. exported, mainly to Germany, France, and England (in the order named).

The cellulose or chemical wood-pulp industry prospered in 1919, in spite of Canadian competition; 15 per cent. of the sulphite pulp and over 35 per cent. of the sulphate pulp were shipped to the United States. Almost all the exported material is produced by the members of the Finland Cellulose Association (Finska Cellulosa Föreningen), which comprises 18 sulphite mills (capital £8,000,000, annual capacity about 150,000 t.), and 6 sulphate mills (capital £4,000,000 and capacity 66,000 t.). The annual report of the association gave the production figures for 1919 as 68,900 long tons of sulphite pulp and 26,870 t. of sulphate pulp. Exports were as follows (long tons):—

Chemical Wood Pulp.	1919.	1913.
Wet .. ..	2,850	3,125
Dry .. ..	69,804	72,728
Total .. ..	72,654	75,853

Russia was the most important foreign market for Finnish paper until 1917, but when this was closed considerable difficulty was experienced in securing new markets owing to Swedish and Norwegian competition. Trade revived in the latter summer of 1919, and increasing quantities have been marketed, particularly in England and the United States. The capacity of the 31 mills represented in the Finska Pappersbruksföreningen is estimated at about 220,000 tons, and their combined capital is over £10,000,000 (at par); the 1919 output was about 95,000 tons, and the sales 66,970 t., of which 44,800 t. was exported.

The metal industries have recently shown some recovery; production has increased by about 30 per cent. over that of 1918 and has reverted to its main object, viz., that of supplying the country with machines, tools, and metal goods. The newly instituted eight-hour day is regarded with misgiving, but as it has also been adopted in Scandinavia and other countries, it is hoped that the industry will not be seriously affected.

Finland has two important Portland cement factories, with a combined annual capacity of 675,000 barrels, but this will soon be doubled; these factories also make lime for use in the cellulose factories. Coal is needed for this industry.

Raw materials having become available, the cotton factories resumed full work in the latter half of 1919, and the prospects of the industry are considered bright. It is stated that the textile manufacturers are very dissatisfied with the way they are treated by the German dyestuff firms, and that there is a good opening for other foreign dyes of good quality.

The manufacture and production of matches could become a very important industry, as 90 per

cent. of the raw materials, notably poplar wood, is indigenous. Production was increased 10 to 15 times during the war; work was suspended when export to Russia was stopped, but resumed in 1918. The Swedish match trust has recently obtained control of the two largest factories and purchased a third. Exports of matches were 8.5 tons in 1913, 290 t. in 1918, and 855 t. in 1919.

The tar industry, formerly important, has declined greatly, production and export having become very small; the quantity of tar exported in 1918 was 629 galls., as against 13,636 galls. in 1913.

The six sugar refineries were combined in January, 1919, into one limited company (Finska Socker), with a paid-up capital of 50 million marks (about £2,000,000 at par) and a total annual capacity of 60,000 tons of sugar. Sugar-beet cultivation has commenced on a small scale, and a factory began work in February, 1920.

Imports into Finland in 1919 were valued at £32,537,485 (£1=75.4 Finnish marks), and included:—Drugs, £117,252; gums, resins, etc., £535,150; oils and greases, £2,636,300; perfumes, £23,213; minerals and manufactures, £632,997; chemicals, £295,170; explosives, £70,961; colours and dyes, £181,698; and metals and manufactures, £2,271,067. Of the total imports, the United Kingdom supplied 27 per cent., followed by the United States with 25 per cent. The exports in 1919 were valued at £11,338,468, of which 43 per cent. was shipped to the United Kingdom.—(*U.S. Com. Rep. Suppl.*, Nov. 11, 1920.)

## LEGAL INTELLIGENCE.

### AGENCY CLAIM AGAINST A COMPANY MANUFACTURING SALICYLIC ACID. *E. T. Pearson and Co. v. Sheppard's Chemical Works, Ltd.*

In the Chancery Division, on February 9, Messrs. E. T. Pearson and Co., of London, made an application that the liquidator of Sheppard's Chemical Works, Ltd., should be directed to admit their claim for £3918 as creditors of the latter company.

For the applicants it was stated that they were appointed sole agents for the sale of the salicylic acid manufactured by Sheppard's, and, as the agency was for a term of seven years commencing in June, 1916, it was claimed that the liquidation of the company in June, 18 constituted a breach of contract. The applicants had earned over £1400 in commission on sales, and there was evidence that the sale was increasing.

On behalf of the liquidator it was stated that Sheppard's, Ltd., had not manufactured salicylic acid since it went into liquidation and was re-constituted as a new company, as the chemical could not be sold at a profit, the price having dropped from 22s.—23s. per lb. in 1916 to 2s. per lb. just before the company ceased operations. It was contended that the clause providing for the payment of £1000 when the agreement was ended was not a measure of damages.

Mr. Justice Astbury said that when there was a seven years' agreement it was not an answer to a breach of contract to say that the chemical could not be produced at a profit; the applicants, however, could not get more than the £1000 payable if the agency did not run the full term.

The parties finally arrived at a settlement by which the applicants, who held a sum of £273 for goods sold, were to retain £150 in full satisfaction of their claim and costs, the remainder to be held by the liquidator as a sum out of which the applicants would be indemnified against any claim brought against them in respect of bad quality of goods sold under the agency agreement.

## OFFICIAL TRADE INTELLIGENCE.

(From the Board of Trade Journal for February 10 and 17.)

### OPENINGS FOR BRITISH TRADE.

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W. 1, from firms, agents, or individuals who desire to represent U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number:—

Locality of Firm or Agent.	Materials.	Reference Number.
British W. Indies	Galvanised sheets .. .. .	223
"	Glassware, crockery .. .. .	224
Canada .. .. .	"Stainless" steel, patent medicines .. .. .	191
"	Pig iron .. .. .	193
"	Indian linseed oil .. .. .	"
"	Varnish .. .. .	"
South Africa .. .. .	Roofing (felt and other), rubber, paper, vulcanised fibre .. .. .	222
"	Tinplate .. .. .	222a
Belgium .. .. .	Glass, earthenware, porcelain .. .. .	209
France .. .. .	Chemicals and raw materials, especially for the soap and perfumery industries .. .. .	228
Italy .. .. .	Lubricating oils, paint, varnish .. .. .	207
Latvia .. .. .	Shellac, gum sandarac, gums, resins, ceresin, montan wax, gilsoutite, alginol .. .. .	230
Spain .. .. .	Tinplate, heavy chemicals, paper .. .. .	209
Morocco .. .. .	Glassware, china .. .. .	235
Argentina .. .. .	Metals, chemicals, soap, dyes, candles, tinplate .. .. .	241
Uruguay .. .. .	China, earthenware, glassware .. .. .	211
United States .. .. .	Chemicals .. .. .	240

\* The High Commissioner for Canada, 19, Victoria Street, London, S.W. 1.

**MARKET SOUGHT.**—A Canadian firm manufacturing oak and hemlock sole leathers wishes to get into touch with importers in the U.K. [Inquiries to the Canadian Government Trade Commissioner, 73, Basinghall Street, London, E.C. 2.]

### TARIFF, CUSTOMS, EXCISE.

**Antigua.**—The revised rates of export duties on sugar, syrup, and molasses, with effect from January 1 to December 31, are given in the issue for February 10.

**Austria.**—Among the articles for which import licences are still required are sugar, malt, honey, paraffin, alcoholic beverages, vinegar, opium, essential oils, benzol oils, mineral oils, artificial silk, certain kinds of glass and glassware, stone-ware, asbestos wares, earthenware, certain metals and wares, salt, many chemicals, varnish, perfumery, candles, and soap.

Articles for which export licences are still required include sugar, molasses, oil seeds, raw hides and skins, fats and oils of all kinds, vinegar, pyrites, certain ores, chalk, magnesite, opium, camphor, dyeing and tanning materials, gums, resins, mineral oils, tar, vegetable fibres, paper pulp, paper, rubber waste, cement, certain metals and wares, caustic potash and potassium salts, caustic soda, Glauber's salt, white lead, lead oxides, ammonia, and many chemicals.

**Belgium.**—Margarine and vinegar may now be exported without licence.

**British India.**—The revised Tariff Valuations are set out in the issue for February 17. They supersede those dated November 20, 1919, and take effect as from January 1.

Among the articles admitted duty free are hops, salt (under certain conditions), raw hides and skins,

oil-seeds (under certain conditions), raw wool and cotton, wood pulp, quinine, trade catalogues, and certain substances when used for the weaving of cotton or the baling of cotton goods, e.g., aniline blue, sodium sulphates, sodium carbonate, magnesium chloride and sulphate, zinc chloride, china clay, starch, sizing paste and wax, farina, glutina, and glycerin substitutes.

The export of cinchona bark is prohibited to all destinations except the U.K.

**Bulgaria.**—Revised export duties affect, *inter alia*, skins, hair, edible fats, certain oil-seeds, opium, rose oil, animal waste, scrap metals, paper and rubber wastes.

**China.**—A surtax of 10 per cent. of the existing customs duties will be levied as from March 1.

**Egypt.**—Sesame can now be exported without a special licence.

**France.**—The export duty on dephosphorisation slag is fixed at 1.50 fr. per 100 kg.

**France and Algeria.**—The "coefficients of increase" on calcium carbide, glass, paper, and cardboard have been modified.

**French Colonies.**—The export of sugar, crude gold, platinum and silver is prohibited to all destinations except France and French colonies.

**Germany.**—A list showing the export taxes leviable under section 6 of the Ordinance of December 20, 1919, may be seen at the Department, 18, Queen Anne's Gate, London, S.W. 1.

The import of lard, goose-grease, beef marrow and other fats similar to lard (but not margarine) is permitted without licence as from February 15.

**Grenada.**—An additional export duty has been levied on cocoa under certain conditions.

**Italy.**—Import may be authorised under certain conditions of certain glasswares, laboratory glassware, and photographic films.

**Kenya.**—It is proposed to allow free of customs duty the import of drugs, medicines and rectified spirit to be used solely in the compounding of drugs and medicines.

**Malta.**—Paraffin and stearin may now be imported duty free.

**Palestine.**—The import of explosives, nitre, salt, and "hashish" is prohibited.

The import of cocaine, sulphonal, pierates, and potassium and sodium chlorates is permitted only under special licence.

**Portugal.**—The full text of the Bill which provides for the complete revision of the customs tariff is set out in the issue for February 10. It is proposed to abolish, with a few exceptions, the surtaxes on import duties. The customs duties will be collected solely in gold. The proposed new schedule of duties covers practically all classes of goods.

**Sweden.**—Export prohibitions have been removed from peat, lard, tallow, milk powder, dripping, raw rubber, gutta-percha and balata, caustic potash, potassium carbonate, chloride and sulphate, tartar emetic, salts of gold, platinum and radium, chlorides of tin, indigo, and unsweetened condensed milk.

**Switzerland.**—Export licences are still required for cocoa, chocolate, raw hides and skins, glue stock, fibres for the manufacture of paper, cotton waste, porcelain insulators, raw iron and wares thereof, copper ore, copper, gold, silver, platinum, potassium permanganate, saccharin, methyl alcohol, indigo and aniline, anthracene, naphthalene and tar colours not specially mentioned in the general tariff.

**Tunis.**—Essence of turpentine, and bauxite, may only be exported with the sanction of the Director-General of Finance.

**Uruguay.**—It is proposed to levy an additional duty of 15 per cent. on all imports, but not on certain oils, sugar, kerosene, charcoal, and a few other substances.

## GOVERNMENT ORDERS AND NOTICES.

**OVERSEAS TRADE (CREDITS AND INSURANCE) ACT, 1920.**—By an order of the Board of Trade of February 17, 1921, the country of Austria was added to the Schedule to the Act.

**DECONTROL OF COAL.**—The Board of Trade has notified, under date February 14, that the Directions issued August 25, 1920, relating to maximum pithead prices for coal sold for home consumption and for bunkering vessels will cease to have effect as from March 1, 1921.

**PROHIBITED EXPORTS.**—The Board of Trade (Licensing Section) announces that, by Order-in-Council, dated February 7, 1921, the export of:—Sulphate of ammonia; superphosphate of lime; basic slag; and compound fertilisers containing any of the foregoing substances is now prohibited under the Fertilisers (Temporary Control of Export) Act, 1920; and the Proclamation of May 10, 1917, as amended by subsequent Orders of Council, is revoked so far as it relates to:—Sulphate of ammonia and mixtures containing sulphate of ammonia; basic slag; manures, compound, containing either sulphate of ammonia, superphosphate of lime or potash; superphosphates. Applications for export licences should continue to be addressed to the Licensing Section, Board of Trade, Great George Street, Westminster, S.W. 1.

**DETERMINATION OF COMBUSTIBLE MATTER IN COAL DUST AND MIXTURES THEREOF.**—The Secretary for Mines has issued notices under the Coal Mines Act, 1911, concerning (a) the determination of combustible matter in dust mixtures which contain carbonates (Feb. 14), and (b) the testing of dust mixtures which contain such a high percentage of moisture that they cannot be sieved in the manner prescribed by the Regulations (Feb. 7).

(a) The sample is ignited in an open vessel to decompose completely the carbonates until the weight is constant; the percentage loss in weight is determined by treatment with acid; and the difference between the percentage losses so obtained is taken as the percentage of combustible matter.

(b) The sample, having been well mixed, is dried at 212° F. until the weight is constant, and the percentage of combustible matter is determined as under (a).

**DYESTUFFS LICENSING ADVISORY COMMITTEE.**—In accordance with the provisions of Subsection (3) of Section 2 of the Dyestuffs (Import Regulation) Act, 1920, the President of the Board of Trade has appointed the following Committee to advise the Board of Trade with respect to the granting of licences under the Act:—Mr. Vernon Clay (Joint Managing Director, Robert Clay, Ltd.); Mr. G. W. Currie; Mr. G. Douglas (Managing Director, Bradford Dyers' Association Limited); Mr. E. V. Evans (Hon. Treasurer, Society of Chemical Industry); Dr. M. O. Forster (Director of the Salters' Institute of Industrial Chemistry); Mr. C. C. Railton (Director, Calico Printers' Association, Ltd.); Mr. H. B. Shackleton (Messrs. Taylor, Shackleton and Co., Shipley); Mr. T. Taylor (Cornbrook Chemical Co., Stockport); Mr. S. A. H. Whetmore (British Dyestuffs Corporation, Ltd.); and Mr. W. J. U. Woolcock (General Manager, Association of British Chemical Manufacturers).

Pending the appointment of a permanent chairman, which it is hoped to make at an early date, Mr. P. Ashley, Assistant Secretary, Industries and Manufactures Department, Board of Trade, will act as chairman of the Committee. The Secretary to the Committee is Mr. W. Graham, and all applications for licences should be addressed to the Secretary, Dyestuffs Licensing Committee, Danlee Buildings, Spring Gardens, Manchester.

## COMPANY NEWS.

### SOUTH METROPOLITAN GAS COMPANY.

At the annual meeting held on February 9, the chairman, Dr. Charles Carpenter, stated that the company's Act of Parliament (*cf.* J., 1920, 310 *n*) authorised the payment of a dividend of 5 per cent. per annum on the issued ordinary stock (6 per cent. on future issues), and a dividend at this rate was now proposed in respect of the past year. The increase in expenditure, of which salaries and direct wages accounted for about 40 per cent. and coal for over 46 per cent., had been met by enhanced profits on the sale of residuals. The production of therms for the year was 70·45 per ton of coal carbonised, and there had been an increase of 5·43 per cent. in the sales of gas. Great trouble was still being experienced in obtaining clean coal, but with the passing of Government control an improvement in quality was to be expected. It was to be hoped that the pre-war practice of exporting much of our best coking coal would not be revived; recovery and marketing of the by-products could be done here quite as well as in Germany, and this course would be followed by great economic advantages to this country. Dye-intermediates were now being manufactured on an important scale, and the company was producing successfully a grade of ammonium sulphate equal to that produced in Germany; it was, however, to be regretted that the export of this material had been placed for two years in the hands of a trust. Given time and encouragement, our chemists could learn dye-making from the Germans, just as the latter had learned ship-building from us. Most unfortunately, the Government had not prevented the country being flooded with German dyes, of which £1,831,275 worth was admitted during November last; and it was to be hoped that the heat-resisting glass industry would fare better. It was proposed to promote a Bill in Parliament to make permanent the company's powers to raise capital authorised by the Acts of 1916 and 1918. Notwithstanding the fact that no co-partnership bonus had been paid for the past three years, the employees continued to work in full accord with the management.

## TRADE NOTES.

### FOREIGN.

**The Vegetable Oil Industry of Marseilles.**—This industry has of late experienced many vicissitudes, but the outlook on the whole is now brighter. In Marseilles there are 30 seed-crushing firms owning 48 mills and 2011 presses. The import of oil-seeds, normally about 60 per cent. of the total imported into France, and consisting chiefly of sesame, groundnuts and copra, amounted to 321,354 metric tons in 1919, and that of vegetable oils 78,438 t., compared with 589,597 t. and 40,927 t., respectively, in 1913. Olive oil is only of secondary importance to the industry. The French production, about 6000 t. per annum, is not equal to the demand, and supplies are imported from North Africa, Spain and Italy (*cf.* J., 1920, 217 *n*); the establishment of co-operative mills has proved beneficial. The exports of oil-seeds, oil, and oil-cake from Marseilles in 1919 were 9031 t., 3298 t., and 747 t., respectively, compared with 39,600 t., 52,055 t., and 132,837 t. in 1913. Coconut butter is manufactured on a large scale by four companies, which produced an average of 60,000 t. yearly before the war. Since then this industry has suffered from lack of raw materials and diminished export trade due to the development of the industry in other countries, particularly the United Kingdom, the exports having decreased from 19,510 t. in 1913 to 721 t. in 1918.—(*U.S. Com. Rep.*, Dec. 20, 1920.)

**The Dye Trade in Holland.**—The future of the dye trade in Holland is uncertain, production is small, being mainly confined to three firms, and the textile trade complains that the domestic supply cannot be depended upon. Dyes are therefore being obtained from Germany, although Great Britain, Switzerland and the United States are furnishing an increasing supply; British and American dyes, however, have been criticised for lack of variety. The imports during the first eight months of 1920 amounted to 1825 metric tons (worth £795,330 at par), compared with 1318 t. in the same period of 1918, and included:—Aniline dyes, 1311 t.; other synthetic dyes, 5 t.; synthetic indigo, 350 t.; natural indigo, 5 t.; madder and natural dyes, 84 t.; and dyewood extract, 70 t. About 85 per cent. of the aniline dyes came from Germany, 8 per cent. from Switzerland, and the remainder from Great Britain and America; practically all the indigo and madder came from Germany and the dyewood extract from France.—(*U.S. Com. Rep.*, Dec. 2, 1920.)

**Foreign Company News.—Japan.**—Five celluloid companies, the Chigusa Celluloid K.K., Royal Celluloid Co., Kazushiro Celluloid K.K., Juzen Shokai, and Hata Shokai, have combined to form a new company, the Central Celluloid Co., with a capital of 2,000,000 yen (£204,166 at par), of which 1,400,000 yen is paid up.

A company with capital of 1,000,000 yen is to be formed by the Japan Chemical Industrial Co. and other fertiliser manufacturers to develop the phosphate deposits in Hirata, an island south of Hainan Island.

The Imperial Gunpowder Industrial Co., established in November, 1919, with a capital of 10 million yen, to manufacture gunpowder, explosives, chemicals, and dyestuffs, will probably start production in March, but instead of making mining explosives it will manufacture for the naval department.—(*U.S. Com. Rep.*, Dec. 21, Jan. 25, 1921.)

**Italy.**—A new company, the Società Generale dei Colori, with a capital of 5 million lire and power to extend it to 50 million lire, has been formed at Milan to manufacture mineral colours, thus introducing a new industry into Italy.—(*Z. angew. Chem.*, Jan. 28, 1921.)

**Sweden.**—It is reported that Lever Brothers, Ltd., will shortly dominate the whole Swedish soap industry by acquiring control of seven soap firms, the combined capital of which is about 10½ million kronor (about £590,000 at par). Seven per cent. preference shares in a holding company will be

given in exchange for shares in the Swedish companies.—(*Z. angew. Chem.*, Dec. 17, 1920.)

#### BRITISH.

**Uganda in 1918-19.**—In view of the demands made upon this Protectorate by the war, the results achieved during 1918-19 were quite satisfactory, but as new capital has not been available since 1914, industrial enterprise has been restricted to its former channels. Cotton is the chief product; the value of the crop in the year under review was £965,951, and the area under cultivation, now being rapidly extended, was nearly 150,000 acres. Para rubber was grown over an area of 11,255 acres, and production is increasing yearly; 113 tons was exported. Ceara and Funtumia rubber do not repay exploitation. The production of oil-seeds could be extensively developed; only small quantities of groundnuts, castor seed and sim-sim are at present obtained. The forests of Uganda would well repay exploitation.—(*Col. Rep.—Ann.*, No. 1054, Dec., 1920.)

**Zanzibar in 1919.**—The most important industry in Zanzibar is the cultivation of cloves, which was begun in 1818 and now covers an area of 52,000 acres, with 4,700,000 trees in bearing in the islands of Zanzibar and Pemba. The output averages 6250 tons, heavy crops being produced every 3 or 5 years; the 1918-19 crop was 12,946 t., the largest on record. Coconut cultivation ranks next in importance, and the 48,000 acres planted to this crop could be considerably extended. The copra is badly prepared and compares unfavourably with the Ceylon product; 14,177 t. was exported in 1919. Rubber is obtained from *Landolphia Kirkii*, but there was no crop in 1919; the cultivation of the Ceara rubber tree, attempted unsuccessfully in 1907, has been abandoned. Other crops grown include rice, sugar, areca nut, cassava, chillies, castor plant, kapok, cinnamon, etc. Three soap factories have a combined output of 80 tons a month; the coir fibre, rope and matting industry is found in the coconut districts; and a certain amount of pottery is produced. The value of imports decreased from £2,366,390 in 1918 to £1,934,169 in 1919, and that of the exports increased from £2,133,597 to £2,444,011, cloves and copra being largely responsible for the increment.—(*Col. Rep.—Ann.*, No. 1052, Dec., 1920.)

**Foreign Trade of the United Kingdom in 1920.**—The following statistics show the value of the foreign trade of the United Kingdom in chemicals and related products during the year 1920, based upon the pre-war values of 1913:—

	IMPORTS RETAINED.					EXPORTS OF U.K. PRODUCE				
	Declared Values.		Values in 1920 estimated at average values in 1913.	Percentage excess of average values in 1920 over those of 1913.	Percentage deficiency (—) or excess (+) of quantity in 1920 compared with those of 1913.	Declared Values.		Values in 1920 estimated at average values in 1913.	Percentage excess of average values in 1920 over those of 1913.	Percentage deficiency (—) or excess (+) of quantity in 1920 compared with 1913.
	In 1913.	In 1920.				In 1913.	In 1920.			
Chemicals, drugs, dyes and colours ..	£1,000	£1,000	£1,000	Per cent.	Per cent.	£1,000	£1,000	£1,000	Per cent.	Per cent.
Oils, fats, and resins, manufactured ..	11,926	29,959	8,847	238.6	— 25.8	19,533	40,730	13,505	201.6	— 30.8
Leather and manufactures thereof ..	13,350	70,516	19,704	257.9	+ 47.6	4,444	13,616	4,445	208.3	+ 0.0
Paper and cardboard ..	9,501	17,247	4,746	263.4	— 50.0	5,279	11,673	4,632	152.0	— 12.3
Earthenware, glass, abrasives, etc. ..	7,415	29,747	8,067	268.7	+ 8.8	3,679	12,818	3,512	265.0	— 4.5
Iron and steel and manufactures thereof ..	5,179	10,846	3,178	241.3	— 38.6	7,427	18,324	6,300	190.9	— 15.2
Non-ferrous metals and manufactures thereof ..	15,551	28,198	7,348	283.8	— 52.7	55,351	128,943	40,267	220.2	— 27.3
	21,349	31,069	17,952	73.1	— 15.9	12,036	25,868	14,750	75.4	+ 22.5

—(*Bd. of Trade J.*, Jan. 27, 1921.)

## REVIEWS.

**ORGANIC MEDICINAL CHEMICALS.** By M. BARROW-OLIFF and FRANCIS H. CARR. *Industrial Chemistry Series*, edited by S. RIDEAL. Pp. xvi. + 331. (London: Baillière, Tindall and Cox. 1920.) Price 15s. net.

In taking stock of the literature dealing with chemical industry, it is somewhat remarkable that the oldest branch, that relating to the manufacture of drugs, should be so scantily represented. One reason is undoubtedly the fact that the subject is complicated, an immense variety of substances of different classes having to be considered; another, that the operations performed are on a relatively small scale and perhaps hardly appeal so much to the technical man as the larger industries, heavy chemicals, tar distilling, or even the manufacture of dyestuffs. Indeed, when one deals with many of the synthetic drugs, one is forcibly reminded that their economic manufacture should be carried out in conjunction with the dye industry; the very trade-mark "Aspirin" is the property of the Farbenfabriken vormals Friedrich Bayer and Co.

The appearance of the work now under review is all the more welcome since its authors have been successfully associated with some of the largest British works engaged in the manufacture of drugs, whilst their reputation as scientific chemists is such as to guarantee the absence of slavish reprinting of antiquated processes or the unqualified acceptance as gospel of all the statements made in patent specifications.

One paragraph in the authors' preface may well be commended to those engaged in the industry, namely, that relating to the supply of important synthetic chemicals during the war and the lack of economic basis on which such supply was established. In many cases this could not be helped at the time, but now that the war is over and the desire to retain the young industry in the country is a very real one, "it is of outstanding importance to perfect processes so that the most economical methods are used and the best possible yields obtained." If the directors of chemical firms were generally as well-versed in chemical knowledge as the authors, the desirable result would be attained more rapidly.

The Introduction, occupying only six pages, is apt and interesting. The subject matter is divided into sections arranged according to the uses of drugs rather than to their chemical relationships. This division has many advantages, but it cannot always be strictly adhered to, as is apparent when we turn to the natural alkaloids.

Narcotics and General Anæsthetics form the subject matter of the first section. Ether is dealt with at considerable length, and a good account is given of the manufacture as carried out at H.M. Factories at Pembrey and Gretna. Several diagrams illustrate the beautiful Barbet plant which succeeded in giving 94.3 per cent. of the theoretical yield during a period in which over 5000 tons of ether was manufactured. Following ether, we find methylal, paraldehyde, acetophenone and the simpler halogenated aliphatic compounds dealt with, whilst the section concludes with an account of the sulphone hypnotics and urea derivatives such as adalin and veronal.

Section II., dealing with Naturally Occurring Alkaloids and their Derivatives, is illustrated by several diagrams and is followed by Section III., in which an account is given of Natural and Synthetic Local Anæsthetics: one could wish that more than 24 pages could have been spared for an account of the interesting and varied compounds included. Antipyretics and Analgesics follow in Sec-

tion IV., and here, as in the preceding section, one is struck by the diversity in structure of the compounds used to produce very similar physiological results.

In the case of Organic Antiseptics and Disinfectants (Section V.), phenol, other phenolic compounds and derivatives are taken first. The order followed is rather difficult to understand, for tribromophenol is separated from other phenolic compounds by an account of substances of the type of Chloramine-T: presumably the idea is to keep halogenated compounds together. After formaldehyde and hexamine, certain dyestuffs-antiseptics are dealt with, and the section finishes with the consideration of tannic acid and santalol derivatives.

Purgatives form the subject of Section VI., and Vaso-Constrictors and Vaso-Dilators of Section VII., a considerable amount of attention being paid to synthetic tyramine, histamine and adrenaline. Section VIII., dealing with Diuretics and Uric Acid Solvents, will prove very interesting to many organic chemists even if the subject is not as topical as that of the next section (IX.), viz., Organometallic Compounds.

Section X. gives an account of the digitalis group, skin irritants, glucosides and neutral principles, whilst pituitary and thyroid extracts, vitamins and other substances of interest, not finding any other place in the book, are dealt with in the eleventh and concluding section.

In a new edition, one or two rectifications will doubtless be made. On p. 188, Proflavine is described as 3:6-diamino-acridine sulphate, and on p. 190 Acriflavine is oriented 2:6. According to the system adopted in numbering the acridine ring, both should be 3:6 or 2:8, though the latter is now more commonly used. On p. 294, thiosinamin is referred to in brackets as allyl thiocarbamate, whilst the formula given is that of allyl thiocarbamide.

The book is written in an interesting manner and is well indexed; the printing and diagrams are clear; and as a guide to a good general knowledge of organic medicinal chemicals the work may be warmly recommended.

J. T. HEWITT.

**THE YEASTS.** By A. GUILLIERMOND. *Translated and revised by F. W. TANNER.* Pp. 424. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1920.) Price 33s. net.

In 1912 Professor Guilliermond contributed to the Section of Cryptogamic Botany of the *Encyclopédie Scientifique*, edited by Dr. Toulouse, a volume entitled "Les Levures," which forms the basis of the work under review.

So great has been the progress made in the domain of microbiology during recent years that Dr. Tanner of the University of Illinois, who had undertaken the English version, realised that it would be necessary to revise that which had been written, and to add much new matter, in order to produce a book adequate to the needs of those for whom it was originally intended. From the preface one gathers that the translator has been responsible for the revision of the chapters dealing with the physiological side of the subject, whilst the task of the original author has been that of bringing up to date the chapters on Morphology, Phylogeny and Description of Species.

Prof. Guilliermond is so well known for his investigations in this department of mycological science that his name is a sufficient guarantee that the more purely descriptive part of the subject has been dealt with adequately and well, and such will be found to be the case. The author has, in fact, placed all zymotechnologists (it is to be hoped that the exceedingly objectionable word "fermentolo-



gist" will not be adopted in this country) under a great debt of gratitude, and his book supplies a want which has been long and widely felt for a complete and descriptive list of the yeasts and associated budding fungi.

The system of classification adopted is that of Hansen, with some modifications and extensions. The family of the *Saccharomycetes* is divided into no less than fourteen genera, together with a number of sub-groups, but some of the points of differentiation are very slight and it is doubtful whether such an elaborate system is, in the present state of our knowledge, really justified.

It may be said here that the invariable use, by the translator, of the word "yeast" as the English equivalent of the more general French term "*levure*" is unfortunate, since it is sometimes used correctly to designate the true *saccharomycetes* and at other times, incorrectly, to cover the whole family of budding fungi, including those which do not form ascospores. This is a serious defect and one liable to produce much confusion in the minds of those whose acquaintance with the subject is limited. To such an extent is the misuse of the word "yeast" carried that in the bibliography a paper by the writer of this review is actually incorrectly quoted, the word "yeasts" having been inexcusably substituted for the word "torulae."

Whilst the descriptive portion of this work leaves little to be desired, the more general portion is very unequal and sometimes very disappointing. The plan is ambitious, but one cannot help feeling that too little care has been devoted to the critical selection of material, and the general impression produced by some of the sections is that of a loosely-constructed dictionary article, rather than that of a carefully thought-out contribution to an important monograph. In the section on the influence of oxygen on fermentation, we are told that Pasteur's conclusions have not been invalidated up to the present, and no reference is made to the work of Adrian Brown either in this section or in that describing the use of the "haemocytometer" (haematimeter). In the bibliographical index, however, Adrian Brown's papers on the subject are included, but under the name-heading: "H. T. Brown"!

A good many glaring mis-statements occur in this portion of the book. Thus, on page 3, we meet the truly amazing statement:—"True yeasts never produce endo- or ascospores," whilst, a few pages later, we read of: "the true yeasts producing endospores." On page 163, the word "estimating" should be "detecting," and on page 124 the substitution of the word "alternation" for "attenuation" destroys the sense of the paragraph.

Making all allowance for transatlantic spelling, the translation leaves much to be desired, and errors of spelling are so frequent, especially in the use of proper names, that attention cannot even be called to all those which have met the writer's eye during a cursory survey of the book. The following, however, may be given as instances:—"Leewenhoek," "Mitscherlick," "Boullinger," "Du Claus," and "Schutzemberger;" and among other instances of careless proof-reading, reference may be made to "galactoe," "hydrogen sulfur," "guaiac," "hydriotic acid" and "ciniferin."

The use of the word "levulose" is etymologically unjustifiable, and the use of the word "scnm" for "film" is quite unusual and also less exact. The translator, moreover, uses the word "hydrocarbon" in one whole section, instead of "carbohydrate."

The grammar often leaves much to be desired, and not a few of the sentences are involved and badly expressed.

With all its faults, however, both of omission and commission, the book will be of great value to the

fermentation expert, as well as to the student of general mycology. It is because it is likely to be so valuable and because it possesses such excellent features that the reviewer has felt the necessity of calling attention to defects which can easily be remedied in future editions, and which at present detract somewhat seriously from the usefulness of an otherwise admirable publication.

A. CHASTON CHAPMAN.

THE CHEMISTRY OF ENZYME ACTIONS. By K. GEORGE FALK. *American Chemical Society Monograph Series*. Pp. 136. (New York: The Chemical Catalog Co. 1921.) Price \$2.50.

THE unanimous desire of Anglo-Saxon chemists to emancipate themselves from the influence of German chemical literature and its subtle propaganda has caused a good deal of thought and work to be devoted to the development of chemical literature in the English language. In this connexion special reference may be made to the praiseworthy pioneer efforts of Mr. F. W. Attack and his associates, which at least have had the practical result that the question was one of the subjects of discussion at the Inter-Allied Conference of Pure and Applied Chemistry at its meetings in London and Brussels in July, 1919. It was then agreed that the American Chemical Society, which, it may be remarked, is the sole body representing American chemists, should undertake the preparation of scientific and technological monographs.

With commendable celerity, in which one may be allowed, perhaps, to trace the influence of such hustlers as Dr. C. Herty and Dr. C. L. Parsons, the first of these volumes has now been produced in barely eighteen months from the inception of the scheme. The necessary arrangements for publication have been in the hands of certain gentlemen acting as trustees, and, in particular, of William A. Noyes and John Johnston, acting as editors respectively of the two series. The reviews are intended to present the chosen topic in a readable form intelligible to the general chemical reader who may be engaged in other lines of research, and at the same time to stimulate further research in the particular field. They are thus on the same lines as the monographs originated some years ago by Dr. R. H. A. Plimmer in this country and extended under the progressive auspices of Messrs. Longmans, Green and Co. It is generally admitted that the work done by the writers of the British monographs has been of the highest value to their fellow-chemists, and we may expect the same result from the labours of our American colleagues. Chemical science is to-day so vast, and its literature so complex, that no text-book of the old type can deal adequately with it, and it is only by the study of suitable monographs that the advanced student of the present day can gain the necessary all-round acquaintance with his subject. At a time when so much is made of specialisation it is worth while inserting a special plea for the necessity of the widest possible reading of chemical literature, so as to gain a sound knowledge of the subject. No one of us knows at the end of his university career in which particular sections of chemistry his life-work will be planned, and it is only by the broadest possible reading that the necessary critical faculties can be trained.

Dr. Falk's own work on enzyme action is well known and guarantees his competence to inaugurate the series. Broadly speaking, enzymes may be considered from three somewhat different points of view—chemical, biological, or mathematical, using this term to connote, in the author's words, "the better understanding of the fundamental chemical relations underlying an exact knowledge of chemical reactions"; it is mainly on these lines that the review is compiled. Accordingly,



the first part of the work deals with the more recent theories of chemical structure, the velocities of chemical reactions and their general theory, before specific reference is made to certain reactions catalysed by enzymes. Sections on the physical and chemical properties of enzymes lead up to a discussion of the chemical nature of certain enzymes and the mechanism of their action. A final section is devoted to their uses and applications. The whole work forms a somewhat abstruse review of present-day physico-chemical theories and will repay the closest study by those interested. It obviously invites criticism in detail, for which this is not an appropriate medium. Sufficient has been said to indicate the scope of the work and to wish the scheme every possible success on behalf of the English-speaking chemists on this side of the Atlantic.

E. F. ARMSTRONG.

## OBITUARY.

### WILLIAM ODLING.

The death of Professor Odling, at Oxford, on February 17, in his ninety-second year, removes one who was closely associated with the earliest attempts to lay the theoretical foundations of our science. The name of Odling, together with those of his contemporaries, Williamson, Frankland, Kekulé, Berthelot, Wurtz, and others will live in the history of chemistry for his important contributions to the development of the theory of chemical types, which was originated by Gerhardt, who was inspired by Laurent. Odling also contributed a notable paper "On the Natural Groupings of the Elements" (*Phil. Mag.*, 1857), which contained one of the first attempts to classify elements in accordance with "the totality of their characters." His best-known literary work is his translation of Laurent's "Chemical Method: Notation, Classification, and Nomenclature" (1855), but he was also the author of several text-books and lectures at the Royal Institution and elsewhere. His experimental researches related mainly to the composition of simple organic compounds, the ammonia compounds of platinum, the properties of indium, and aluminium compounds. In later years he investigated various potable waters, publishing in 1886, with W. Crookes and C. M. Tidy, a report to the Huddersfield Corporation on the action of water on lead, and serving as one of the analysts of the London water supply. At the banquet given by the Chemical Society, in 1898, to celebrate the jubilee of six of its past-presidents (among whom was Odling), the president (Prof. J. Dewar) remarked that Odling's work "was characterised by precise and clear ideas, and an almost forensic ability for putting things in a straight, concise, and unembarrassing manner."

Odling was born at Southwark in 1829; he studied medicine and graduated at London University, but work with Gerhardt at Paris led him to take up chemistry, and on returning to this country he became demonstrator in chemistry at Guy's Hospital at the age of 21. In 1850 he was appointed lecturer at St. Bartholomew's, and in 1863 succeeded Faraday as Fullerian professor of chemistry at the Royal Institution. Four years later he was appointed Waynflete professor of chemistry at Oxford, in succession to Sir B. Brodie. His connexion with the Chemical Society dated from 1848, and he served it as secretary from 1856 to 1869, and as president in 1873-74. He was made Fellow of the Royal Society in 1859, and doctor of mathematics and physics by the University of Leyden in 1875.

## PUBLICATIONS RECEIVED.

THE CHEMISTRY OF ENZYME ACTIONS. By K. G. FALK. *American Chemical Society Monograph Series*. Pp. 136. (New York: The Chemical Catalog Co., Inc. 1921.)

THE PHYSIOLOGY OF PROTEIN METABOLISM. By E. P. CATHCART. *Monographs on Biochemistry*, edited by R. H. A. PLIMMER and F. G. HOPKINS. New edition. Pp. 176. (London: Longmans, Green and Co. 1921.) Price 12s. 6d.

A RESEARCH ON THE EUOALYPTS AND THEIR ESSENTIAL OILS. By R. T. BAKER and H. G. SMITH. Pp. 471. Second edition. *Technical Education Series*. No. 24. Department of Education, New South Wales. (Sydney: Government Printer. 1920.)

RAPID METHODS FOR THE CHEMICAL ANALYSIS OF SPECIAL STEELS, STEEL-MAKING ALLOYS, AND THEIR ORES AND GRAPHITES. By C. M. JOHNSON. Third edition, revised and enlarged. Pp. 552. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1920.) Price 36s.

ECONOMIC MINERALOGY. By THOMAS CROOK. Pp. 492. (London: Longmans, Green and Co. 1921.) Price 25s.

THE TECHNICAL EXAMINATION OF CRUDE PETROLEUM, PETROLEUM PRODUCTS AND NATURAL GAS. By W. A. HAMOR and F. W. PADGETT. *Mellon Institute Technochemical Series*. Pp. 591. (New York and London: McGraw-Hill Book Co., Inc. 1920.) Price 30s.

THE REPAIRING OPTICIAN. *Oxford Technical Manuals*. By J. FRAY. Pp. 183. (London: Henry Frowde and Hodder and Stoughton. 1920.) Price 8s. 6d.

REPORT OF THE DOMINION GRAIN RESEARCH LABORATORY. Board of Grain Commissioners for Canada. Department of Trade and Commerce. Pp. 74. (Ottawa: J. de L. Taché. 1920.)

RAPPORT SUR LE COMMERCE ET L'INDUSTRIE DE LA SUISSE, 1919. (Zurich: Union Suisse du Commerce et de l'Industrie. 1920.)

MINERAL RESOURCES OF THE UNITED STATES, 1917. PART II.—NON-METALS. Pp. 1293. *United States Geological Survey, Department of the Interior*. (Washington: Government Printing Office. 1920.)

PUBLICATIONS OF THE UNITED STATES GEOLOGICAL SURVEY. Department of the Interior. (Washington: Government Printing Office. 1920.) 1921:—

STRONTIUM IN 1919. By G. W. STONE.

GYPSUM IN 1919. By R. W. STONE.

LEAD IN 1918. By C. E. SIESENTHAL.

SAND AND GRAVEL IN 1919. By R. W. STONE.

PUBLICATIONS OF THE UNITED STATES BUREAU OF MINES. Department of the Interior. (Washington: Government Printing Office. 1920):—

THE MANUFACTURE OF SULPHURIC ACID IN THE UNITED STATES. By A. E. WELLS and D. E. FOGG. Pp. 218. Price 40 cents.

BIBLIOGRAPHY OF PETROLEUM AND ALLIED SUBSTANCES. 1917. By E. H. BURROUGHS. Price 25 cents.

PRODUCTION OF EXPLOSIVES IN THE UNITED STATES. 1919. By W. W. ADAMS. Price 5 cents.

COKE-OVEN ACCIDENTS IN THE UNITED STATES. 1919. By W. W. ADAMS. Price 5 cents.

THE NATIONAL PHYSICAL LABORATORY. COLLECTED RESEARCHES. Vol. XV., 1920. Pp. 329. (London: H.M. Stationery Office. 1920.) Price 2s.

### DANGEROUS DRUGS.

The hubbub which has arisen in connexion with the Dangerous Drugs Act is an example of the futility of allowing lay officials to deal with a highly technical subject, and has been aggravated by contemptuous refusals to accept the advice of competent authorities. The draft Regulations issued by the Home Office for the working of the Act are so impracticable and display such ludicrous ignorance of the actual conditions obtaining in medicine and pharmacy that they have aroused the ire and scorn of medical men, veterinarians, pharmacists, and the general public alike.

The inception of this Act "to regulate the importation, exportation, manufacture, sale, and use of opium and other dangerous drugs" was due to the need of giving effect to the International Opium Convention signed at The Hague in 1912. By the Treaty of Versailles (Article 295) Great Britain was pledged to bring the Convention into operation within twelve months of the coming into force of the Treaty. There was no serious opposition to the passing of the Act, the principle being accepted by all concerned as wholesome legislation. In the Committee stages of the Bill in the House of Commons several alterations were made mainly as a result of the informed criticism of Mr. W. J. Uglow Woolcock, general manager of the Association of British Chemical Manufacturers, who was formerly secretary to the Pharmaceutical Society, and able to speak with authority.

The Act eventually came into force on September 1, 1920, and besides restricting or prohibiting (except under licence) the import and export of raw opium, prepared opium, and cocaine, it controlled the manufacture, use, and distribution of cocaine, morphine, ecgonine and diamorphine (heroin), and of preparations containing any of these. The real trouble began with the issue, early in January, of the draft Regulations. These were so drawn as to make it illegal to sell without a doctor's prescription such ordinary household remedies as cough mixtures (containing morphine or heroin in medicinal quantities), chlorodyne (containing morphine), or even gall-and-opium ointment. Twenty-five official preparations in the British Pharmacopoeia are thus prohibited, as well as hundreds of unofficial preparations at present in daily use. The Regulations go far beyond the requirements of the International Opium Convention, which expressly recognised existing laws and regulations and laid down that nothing should "apply necessarily to medical prescriptions and to sales made by duly authorised pharmacists." The retail sale of all the drugs mentioned in the Dangerous Drugs Act is already subject to very strict control by qualified pharmacists under the Poisons and Pharmacy Acts. In addition to previously existing restrictions, however (of which the official draftsmen were apparently entirely ignorant), these Regulations provide for five separate books to be kept by pharmacists and four by doctors or veterinary surgeons, in which records of sales, etc., have to be kept.

Pharmacists were promptly up in arms as soon as the Regulations were issued, and when it dawned upon the medical profession that a policeman might enter a doctor's surgery and overhaul his stock of morphine preparations, etc., in a vain endeavour to trace the whereabouts of a minim of hypodermic solution of morphine, and that every doctor would have to keep a separate record of each issue of morphine, cocaine, opium, and diamorphine the medical temper likewise began to rise. Furthermore, when it was discovered that no farmer or stock-owner could keep a colic draught for his

cattle, that no householder could store laudanum, Dover's powder, or gall-and-opium ointment in his medicine cupboard, the outcry against this inept Departmental legislation developed to such an extent that it has induced a gradual and progressive "climbing down" on the part of officialdom. What the ultimate result will be it is difficult to gauge, but the situation at present is that the Home Office autocrats in charge have turned down the commonsense suggestion that the Regulations should be referred to a committee of medical and pharmaceutical experts for consideration as a whole.

### VITAMINS AND THE FOOD SUPPLY.

A. HARDEN.

It is now well recognised that a satisfactory diet must not only contain a sufficient supply of energy along with a provision of protein and mineral substances, suitable both qualitatively and quantitatively for the needs of the organism, but that it must also furnish certain hitherto unidentified accessory factors, the so-called vitamins, required, it is true, only in minute quantity, but indispensable for growth and proper maintenance. Three of these principles have so far been differentiated, and these are most conveniently termed, adopting Drummond's modification of Funk's nomenclature, vitamins A, B and C. The evidence on which the belief in the existence of these three essential dietary factors is based is by this time familiar and can be found in many quarters (see especially Report of the Committee on Accessory Food Factors, Special Report Series No. 38, M.R. Council, and Physiological and National Needs, Constable).

Two important points must, however, be borne in mind throughout any discussion of the vitamin question. First, that the mammalian organism is unable to synthesise these principles for itself and is entirely dependent for its supply on the food with which it is nourished; second, that the young growing organism requires a relatively greater supply of them than the adult. It must further be remembered that the offspring is in its early stages entirely dependent for its nutrition upon the mother, who has therefore to receive a supply of vitamins adequate for the needs both of herself and her young.

The distribution of these factors among the common articles of diet of man and the domestic animals is being gradually worked out and is already known in its general qualitative relationships, but requires a large amount of further quantitative investigation. The table overleaf will serve to remind the reader of the specific importance of each of the vitamins and to give a rough idea of the kind of food material in which it may be expected to be found.

#### Vitamin Requirements.

When we come to consider the vitaminic requirements of an average individual we have to confess to a lamentable lack of accurate information. We are, indeed, in a better position with regard to many of the lower animals, for it has become possible to ascertain with some degree of accuracy by direct experiment the amount of milk or orange juice necessary to protect a guinea pig or a monkey from scurvy, or the amount of yeast which will perform a similar function for a rat or pigeon with regard to polyneuritis. One important conclusion which has been definitely ascertained in this respect is that different animals, even although closely

related, vary very considerably in their need for vitamins. A striking instance is afforded by the guinea pig and the rat, both rodents, in relation to the antiscorbutic vitamin. The guinea pig is very sensitive to lack of this principle and on a scorbutic diet rapidly shows symptoms of scurvy and finally dies of the disease. The rat on a similar diet shows scarcely any sign of the absence of the principle, grows to a good weight and produces young. Careful examination has, indeed, revealed a certain inferiority in the scorbutically fed animals, but scurvy never develops. A less extreme difference is shown by a comparison of the antiscorbutic needs of the guinea pig and the monkey, animals which are both affected by scurvy. The latter, of about ten times the weight of the former, requires almost exactly the same amount of antiscorbutic (orange juice or milk) to protect it from scurvy.

With regard to human requirements we are compelled to turn to a consideration of the actual diets which have been found to keep us in health and draw what conclusions we can from an analysis of these. Except in the case of the very young, these diets are generally of a mixed and varied character so that accurate deductions are difficult. Reference to the table will show which constituent of the daily diet must in most cases be the source of each of the vitamins. As regards the child we are in a somewhat better position. The antiscorbutic requirements (as well as those of vitamins A and B) of a child of 5—7 kilos. are usually considered to be met by a diet of about 500 c.c. of cow's milk. Although, as we shall see, milk is itself a variable quantity, and the above amount is not strictly a minimum, this affords a basis for a rough com-

parison with the monkey and guinea pig, which both require 100 c.c. of milk daily.

#### *Variation in the Vitamin Content of Foodstuffs.*

It is almost certain that the amount of vitamin contained in natural foodstuffs is not a constant quantity, but varies under different conditions of season, age, and so forth. Very little accurate work has yet been done in this direction, but indications have been observed, for example, that the antiscorbutic potency of grass is greater in the young than in the more mature growth. An extreme case is afforded by the well-known production of the antiscorbutic vitamin during the germination of seeds, which themselves contain none, or very little, of this principle.

When we come to deal with secondary foodstuffs—derived from animal sources—a further complication arises, which is well illustrated by the case of milk. The vitamins of the tissues of the animal which is secreting milk are all derived from the foodstuffs of the diet. If these are varied so that very little vitamin is supplied in them several possibilities arise. The supply of milk may be kept up but its vitamin content may be diminished, so that the young suffer, or the vitamin content may be maintained for some time at the expense of the tissues of the mother. On the other hand, the difficulty may be met by a smaller secretion of a milk of the original quality. Here, again, the experimental observations are not conclusive. Hopkins at Cambridge, found that the fat-soluble factor (vitamin A) of goat's milk remained approximately constant when the animal was given a diet deficient in that respect, but that the quantity yielded fell off. On the other hand, in America, several investigators

TABLE I.

	Vitamin A.	Vitamin B.	Vitamin C.
Synonyms .. ..	Fat-soluble A	Water-soluble B Anti-neuritis	Water-soluble C Anti-scorbutic
Effect of deficiency..	Retarded growth ending in death Eye-disease (Keratomalacia) Probably one factor in causation of rickets	Retarded growth ending in death Beri-beri Polyneuritis of birds, rats, etc.	Scurvy
Chief sources ..	Animal fats Fish liver oils Green vegetables Egg-yolk Butter and milk Oleo-margarine, in proportion to animal fat.	Seeds, particularly in the germ, pericarp and aleurone layer Yeast Egg yolk	Green vegetables (especially <i>Cruciferae</i> ) Orange and lemon juice Tomatoes Germinated seeds Swedes and turnips
Present in smaller amounts.	Oil-seeds Carrots	Meat Milk Potatoes and some other tubers and roots Green vegetables Some fruit juices	Meat Milk Potatoes and some other tubers and roots Many fruit juices Dried vegetables
Absent from ..	Most vegetable oils Most lard Most margarine White bread Egg white Yeast Purified proteins and carbohydrates	White bread Polished rice Fats Egg white Purified proteins and carbohydrates	Seeds White bread Fats Yeast Purified proteins and carbohydrates

orking with cows found only a small diminution in the milk flow, but a large depreciation in the antiscorbutic potency of the milk, when the cows were given a diet deficient in vitamin C. Indications have, moreover, been frequently observed of the antiscorbutic inferiority of winter to summer milk, and the winter feed given to cattle in this country is frequently very deficient in both vitamins A and C. At this point laboratory investigations come into close contact with practice, and it should not be difficult to devise a winter feed for cattle which could ensure a supply of milk containing as much of the various vitamins as that produced during the summer. Precisely similar conditions hold for human mothers, and there seems little doubt that lack of sufficient vitamin in the diet of expectant and nursing mothers is often responsible for ill-health or positive disease in their children, such as rickets and scurvy—obvious or incipient—with all their evil and lasting results. It is, moreover, obvious that the vitamin content of many important foodstuffs, such as butter, meat, animal fats, etc., must be dependent on the diet of the animals from which they are derived.

#### *Influence of Processes of Manufacture or Preparation.*

We have so far considered foodstuffs in their natural state, but to the variations inherent in the nature of the different food materials or due to modifications of the diet of animals, must be added those brought about by the treatment which many foodstuffs receive before they reach the consumer. *Milling of Grain.*—The classical example of this is the milling of grain (rice, wheat), in which the germ, or embryo, which contains by far the greater part of the vitamin B and most of the pericarp and aureole layer, which contain the rest, is removed. It is now well known, the unbalanced use of milled wheat leads to beri-beri, and is the main cause of that disease in the tropics. In the same way the British soldiers during the siege of Kut suffered from beri-beri on account of the lack of vitamin B in their diet of white bread and meat, whereas the Indian troops escaped this scourge because their ration included a kind of lentil (rich in vitamin B), and their food was made from a less "perfectly" milled grain. It is interesting to note, in passing, that regarding scurvy the conditions were reversed. In the total absence of raw vegetables and fruits the British were protected by their large ration of tinned food; the Indians, who refused to eat meat, died down with a scurvy.

*Desiccation.*—The effect of drying varies very greatly with the nature of the food material. Cabbage, the richest known source of the antiscorbutic vitamin, when dried, even at a low temperature, loses 70 to 80 per cent. of its antiscorbutic potency, and similar results are given by many other vegetables. On the other hand, fruit juices (orange, lemon) can be evaporated *in vacuo* with little or no loss. About milk some uncertainty prevails. Fresh cow's milk is a very poor antiscorbutic, only about 1 per cent. the value of raw cabbage, but, moreover, as has been already emphasised, it varies in character with the diet of the cow. The potency of dried milk depends in the first instance on the process employed for drying, and there is little doubt that if undue exposure to a high temperature (and possibly to aeration) is avoided, dried milks can be prepared not greatly inferior in antiscorbutic potency to fresh milk. On the other hand, it has been definitely proved by experiments on monkeys (and guinea pigs) that many samples of dried milk actually put on the market contain less of the antiscorbutic principle than fresh milk. So markedly is this the case in some instances that an animal which has acquired scurvy on a certain ration of dried milk has been cured by an equal ration of fresh milk. Vitamins A and B,

on the other hand, are comparatively less affected by desiccation, although here, again, the effect varies with the nature of the foodstuff. Thus, milk appears to suffer very little loss in either of these respects on drying, whereas cabbage loses a large part of its vitamin A on drying, although not so high a proportion as of the antiscorbutic.

*Preservation.*—Comparatively little is known of the effect of preservation on the vitamin content of food materials. The most accurate information available concerns the antiscorbutic factor. Dried cabbage has been carefully tested in this respect by Holst, who has found that when it is air-dried and preserved over phosphorus pentoxide the antiscorbutic potency (which, it must be remembered, is only a small fraction of that of fresh cabbage) is retained even at 37° C. for long periods. When preserved over calcium chloride, however, it rapidly deteriorates, and more rapidly as the temperature at which it is preserved is raised. Dried orange juice, too, has been found to retain its potency over considerable periods, and it seems probable that a perfectly dry powder will withstand preservation at ordinary temperatures for a long time, whereas in the presence of even very small amounts of moisture deterioration will occur. Lemon juice under ordinary conditions rapidly deteriorates, but in presence of the natural antiseptic provided by the oil of the rind has been kept for as long as two years at ordinary temperature, whereas at 37° C. in the same time it became quite inactive.

*Heat.*—The action of heat on the vitamin content of foodstuffs is, of course, of fundamental importance, since many of the processes of food preparation and preservation involve the application of heat at some stage. The experimental investigation of the question has been complicated by the fact, which at first escaped observation, that certainly in the case of the fat-soluble vitamin A, and probably in that of the antiscorbutic C, aeration plays an important part as well as rise of temperature. It has so far been found that vitamin A is moderately stable towards rise of temperature, provided that the presence of air is avoided, whereas in the presence of air it is rapidly inactivated even at comparatively low temperatures. Thus, butter-fat heated at 120° C. in an autoclave for four hours was found still to contain abundance of vitamin A, whereas when it was simultaneously aerated by a current of air it lost the whole of its potency in the same time. This loss on aeration has also been found to occur in six hours at as low a temperature as 50° C. Cabbage retains practically the whole of its vitamin A on cooking, and milk, like butter, can be autoclaved at 120° for an hour without its value in this respect being perceptibly impaired. With regard to the antiscorbutic vitamin, matters are not quite so clear. The effect of heat seems to depend largely on the nature of the material in which the vitamin occurs. Thus, cabbage juice rapidly loses a large proportion (about 85 per cent. of its potency) when heated for an hour at 100° C., whereas swede juice only loses 50 per cent., and orange juice (even when neutralised) is unaffected under the same conditions. Whether aeration is concerned is not yet definitely settled. In milk the antiscorbutic vitamin is certainly less stable than vitamin A, since autoclaved milk is free from antiscorbutic, but contains a considerable amount of vitamin A. Vitamin B (the antineuritic) must be regarded as moderately stable towards rise of temperature, since it has been found to be little affected by heating for 1–2 hours at 100° C. (wheat embryo, yeast extract), and has even been known to survive boiling with sulphuric acid (Funk).

The practical conclusion as regards processes of sterilisation is that at present it is unsafe to dogmatise, particularly with regard to the antiscorbutic vitamin, so that each new case really re-

quires special investigation. It is already known, for example, that the process of canning largely destroys the antiscorbutic value of green vegetables, whereas canned tomatoes retain a large part of the high activity of the fresh fruit.

**Oils and Fats.**—Particular interest attaches to the presence of vitamin A in oils and fats because these are constituents of all diets and, moreover, form in many cases the chief, if not the only, source of this vitamin. Almost all animal fats have been found to contain this substance, the striking exception being lard. As this question has recently been brought before this Society by Drs. Drummond and Zilva, it will suffice to state that this exceptional deficiency has been found by them to be in part due to the processes of extraction and refinement and in part to the peculiar relation of the pig to this vitamin. Fish oils are very rich in vitamin A, but it is thought that much is lost in the processes of refining. Hydrogenation absolutely destroys this vitamin, hydrogenated whale oil, for example, being absolutely devoid of it, whereas the natural oil is rich in it. Whether this is due to the actual reduction, to the catalyst, or to the conditions prevailing during the treatment is not yet known.

The vegetable oils form a problem of great importance in this respect. Most of the oil-seeds contain more or less of the vitamin, whereas many of the vegetable oils as placed on the market, and used for the preparation of margarine, are devoid of it. Whether this is due to the small proportion present in the original seed, to the process of extraction or to the subsequent treatment of the oil is not definitely known. Further investigation is urgently called for to settle these important questions. Much has been already done at the initiative of the manufacturers, and thanks to the wise liberality of the Medical Research Council, which has devoted large sums to the support of investigations on the vitamins, steps are now being taken to make a systematic study of this subject, which has so vital a bearing on the health of the population in general and of the child population in particular.

## FILTRATION BY ROTARY VACUUM-FILTER.

F. BLOOR.

At the Conference on Filtration held last July by the Chemical Engineering Group little attention appears to have been given to filtration by rotary vacuum-filters, although one would gather from the remarks of E. Hatschek (*cf. J.*, 1920, 227 r) that, owing to the accessibility of the cake, whereby both washing and removal are facilitated, and the fact that the filtering process can be made continuous, the method would be one in fairly general use in the chemical industry. However, except for a few products, such as soda ash, produced on a large scale, and its use for extracting salt from ammonium sulphate, the rotary vacuum-filter seems to find little favour at present among technical chemists.

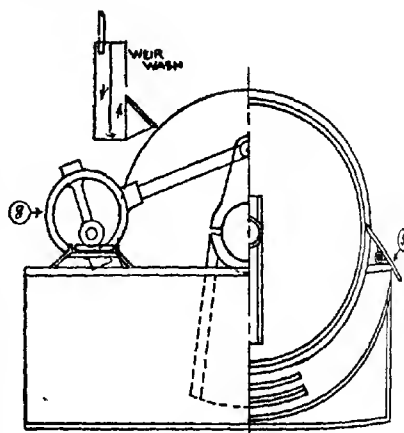
In a recent issue of the *Chemical Trade Journal* (Vol. LXVII., p. 741) a chemical manufacturer writes:—"I have cut my filtration costs down from £3 to 1s. 9d. per ton by the use of continuous automatic filters." Although the type of filter is not stated, the inference to be drawn is that the enormous saving in labour costs has been brought about by the substitution of a continuous filter (such as the rotary vacuum-filter) for a neutch or filter press. It is the possibility of this saving in labour charges and the belief that objections to this type of filter are ill-founded which induce the writer to advocate its more extensive use.

As illustrating the low labour costs which are possible with a rotary filter, one may instance the case of a filter with a drum 6 ft. in diameter by 3 ft. 4 in. wide discharging 6 to 8 tons of soda ash per hour at a labour cost of less than 6d. per ton; moreover, for this cost the material is deposited (by a belt-conveyor) at any desired spot. The question for the chemical manufacturer is, however, whether this type of machine can fulfil the necessary filtering desiderata when dealing with products that differ from soda ash (for instance, in the filtering and washing of sludges, amorphous substances, dyestuff-intermediates, as well as dyestuffs themselves), and can give better results at less cost than neutches or filter presses. In the opinion of the writer this type of filter can be made to deal efficiently with substances of the kind mentioned.

The superiority of the rotary vacuum-filter over other types of filter can obviously only be demonstrated by actual filtration tests on the products concerned, and after comparing filter capacities, costs, efficiencies (washing, etc.). The writer has carried out a few tests with this object in view, but the filter was the ordinary type (described below) without the improvements suggested. However, by measuring the thickness of cake which could be built up and the speed of rotation, it was possible to estimate fairly accurately what a suitably designed rotary filter could do.

The following are actual examples of two very dissimilar products:—(1) A light crystalline substance; cake built up  $\frac{1}{4}$  in. thick, drum rotating 14 revolutions per minute. (2) Sludge (very sticky), cake built up  $\frac{1}{4}$  to  $\frac{1}{2}$  in. thick, drum rotating 1 revolution per minute. This cake gives great trouble in washing in a wash-press, but was washed efficiently on the rotary filter.

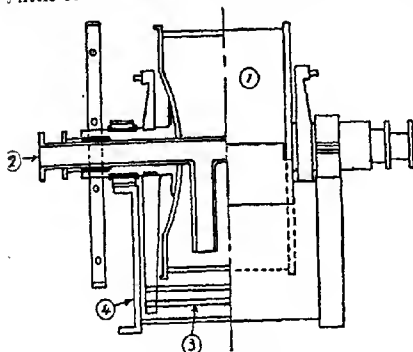
With a filter of the size already indicated it should therefore be possible to filter and wash products similar in nature to the first at the rate of 5 to 6 tons an hour, whereas with sticky material the rate would of course be much slower. The rate for a sludge (which is a difficult substance to filter) would be about one ton per hour.



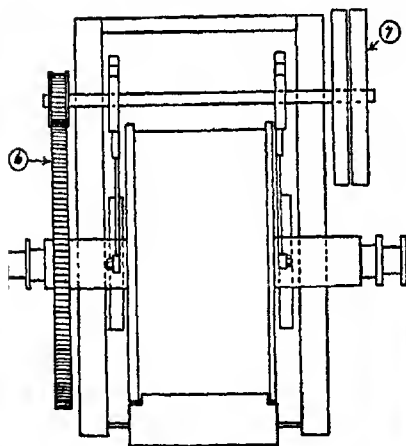
Side view. (Half section.)

The accompanying sketches illustrate an ordinary rotary vacuum-filter without any reverse flow or cloth-cleaning device. The drum is perforated and rotates at a suitable speed (this can only be found by experiment). Over the drum perforations is stretched a suitable filtering medium (cloth or wire gauze), which is secured to the drum by wire or stout cord. The axle of the drum is stationary and forms part of the vacuum system. By producing

vacuum on the inside of the cloth, which is immersed in the reservoir, liquor is drawn through the filtering medium, leaving a film of solid on the cloth. When the drum is rotating this layer of solid can be washed with a suitable solvent (the washings being kept isolated from the filtrate, if necessary). It will be evident that this type of filter lends itself to the use of mechanical devices for drying or steaming the cake and also for dealing with fumes. Where the drum passes the "doctor," or knife, the solid is scraped off, and should then be in a form very suitable for bandling at little cost.



Front view. (Half section.)



Plan.

Rotary Vacuum Filter.

KEY.

- Perforated Drum.
- Vacuum Pump.
- Agitator.
- Trough.
- Knife.
- Gearing.
- Pulley.
- Eccentric (to operate agitator.)

requires very little knowledge of filtration to that the whole success of the filter depends on maintaining the cloth clean; otherwise the rate of filtering will slow down to such an extent as to make the method valueless. In the case of a crystalline body like soda ash the "doctor" is clear of cloth by about  $\frac{1}{4}$  in., so that the cloth requires cleaning before it enters the liquid. This is easily accomplished by causing a portion of the filtrate to pass through the cloth again (usually termed a re-flow). The filter will run continuously for

days with very little attention, but with sludges (e.g., from limed-out sulphonations) the problem is more difficult and cannot be solved as easily as for soda ash. The layer of sludge collecting on the cloth will be very thin ( $\frac{1}{4}$  to  $\frac{1}{2}$  in.), and the "doctor" and drum must be adjusted so accurately that practically all the solid can be scraped off without injury to the cloth. In addition, a very efficient cloth-cleaning device should be used, and the writer suggests that this is best achieved by designing the drum of the filter so as to leave the binding-wire or cord flush with the filtering medium, and providing an internal pressure chamber (designed for varying pressures of air or liquid) to keep the pores of the cloth clean. The absence of these last two factors has caused the rotary filter to be condemned as unsuitable for dealing with such materials as sludges and other products from chemical and dyestuffs factories.

Apart from the question of low labour costs, there is no doubt that the maintenance cost (filter cloths, repairs to power services, etc.) will be lower for a rotary vacuum-filter than for filter presses using compressed air and hand-scraped cloths. However, each filtering process is a problem in itself, and the best and most economical results can only be achieved by experimenting with all the available types of plant. In the past it is doubtful whether chemical manufacturers have given to the rotary vacuum-filter the consideration that its merits deserve.

## AN INTERESTING FIRE IN A LABORATORY.

T. HOWARD BUTLER.

The following account of a fire, which took place in the laboratory of Messrs. Wm. Butler and Co. (Bristol), Ltd., will no doubt be of interest to members of the Society, as the circumstances were extraordinary, and may be of use as a warning to other chemists.

The diagram shows the apparatus that was employed for distilling coal-tar creosote oil. The still is the one recommended by the Ministry of Munitions, and used during the war by its advisors, Dr. H. G. Colman and Dr. P. E. Spielmann, for estimating the amount of benzene and toluene in tar. It will be noticed that the iron still is heated by means of a ring burner which completely surrounds the still, and which may be moved up or down as desired with the object of facilitating the distillation of a watery tar.

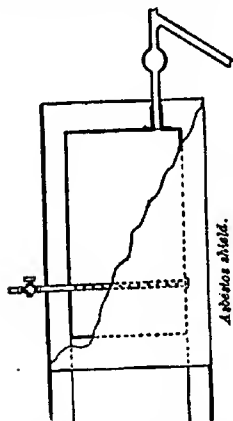
In the distillation in question the still contained about 0.75 gall. of creosote, to which had been added 200 c.c. of a heavy naphtha to facilitate distillation of the small quantity of water in the original creosote. The object of the distillation was to obtain a fraction of creosote oil distilling within a certain range of temperature.

The distillation had been carried out with the ring burner placed about three inches from the bottom of the still, and apparently all the water and naphtha had been removed, as the distillation had ceased. The flame of the ring burner had not been raised, and the contents of the still were "sweating," as it was desired to be sure that all the water had been distilled off. This "sweating" had been going on for some time, approximately one hour, and the temperature of the vapour at the top of the still during this time was  $210^{\circ}\text{C}$ ., which indicated that the temperature of the liquid in the proximity of the burner was probably about  $250^{\circ}\text{C}$ .

At the time of the accident, fortunately, no person was in this laboratory, and the first indica-

tion of any trouble was given by a chemist who was passing the door and saw that the still was "bolting over." This person warned the chemist who was in charge of the distillation, and before either could get into the room, an increasing hissing noise, that can only be described as like a roaring wind, was heard. Both chemists rushed towards the room, which is about 21 ft. long, by 12 ft. broad, by 12 ft. high, and found it completely full of a thick black cloud.

Fortunately this laboratory was fitted with a stop-cock on the gas-main outside the room itself. This was immediately turned off, but there was a small quantity of creosote in a tray in the near vicinity of the still on fire. They removed this, together with some smouldering papers which were on the table in the centre of the laboratory. As the room was completely filled with this black smoke, and all apparent danger had ceased, the laboratory was left for several hours until the carbon had settled. The heat of the combustion or slow explosion, whichever it may be termed, was distinctly felt in the adjoining laboratory, although no serious concussion, beyond the already described "rushing wind" sound, was noticed.



Still, 6 in. diam., 1 ft. high.  
Capacity, about 1 gall.  
Charge,  $\frac{1}{2}$  gall.  
Burner at time of explosion, 5 in.  
from bottom.  
Charge about 5 in. from top.

The room was carefully examined the following day, and it was found that intense heat must have been developed during the accident, as a small part of the lead bench had been melted, and paint on the shelves and beams supporting the ceiling was badly blistered, also note books, papers, and similar articles lying on the table in the centre of the laboratory were completely charred; in fact, the appearance of the laboratory the next day gave the impression that a fire had been burning in the room for some considerable time; as a matter of fact, the actual fire lasted only some few seconds.

An interesting feature of the occurrence was that within two yards of the still there was a bottle containing about 100 c.c. of ether which remained unaffected.

It is not easy to determine definitely the cause of the accident, but it is probable that during the "sweating" process the top portion of the liquid became heated to a fairly high temperature, and that probably there was one or perhaps more drops of water condensed on the top of the still, which fell into this heated liquid and caused the "bolting." This may have become so violent that the cork was blown out of the still and ebullition again increased until there was a considerable cloud of creosote vapour above and around the still; and then this vapour was fired by the flame from the ring burner. It is conceivable that the cloud was in a condition of part vapour and part spray, and that when it caught fire it burned so rapidly that what might be termed a slow explosion ensued.

As the lessons that can be drawn from such an accident are of great value for future precautions, it may be of interest to observe that there is always a danger in heating any vessel to a comparatively high temperature at a point other than the bottom, unless the bottom be also heated; and in this case it seems probable that the accident would not have occurred if a small flame had been playing on the bottom of the still. This would have ensured the whole of the contents of the still being at one temperature. The superheating would probably not have been serious if the ring burner had been set nearer the bottom of the still. It is doubtful if "sweating" should be prolonged for any length of time; if the creosote had been heated more rapidly, immediately after the water had been distilled over, the rush of vapour would probably have brought over any small quantity of water that had condensed on the top of the still. Again, a ring burner should not be used without bottom heating, and it should be rapidly lowered to near the bottom of the still by the time that there are indications that all the water has been distilled over.

The above explanation and the conclusions drawn from it are put forward tentatively in the hope that others who have experienced similar accidents will also communicate their observations and inferences to this Journal.

## SOCIETY OF CHEMICAL INDUSTRY.

### JOINT MEETING WITH THE INSTITUTION OF MECHANICAL ENGINEERS.

A special meeting of the Society was held conjointly with the Institution of Mechanical Engineers—the first, it is to be hoped, of a series of similar meetings—at the Institution, Storey's Gate, S.W. 1, on March 4, when Monsieur Paul Kestner read a paper on "The De-gassing and Purification of Boiler Feed-Water." Capt. Riall Sankey, president of the Institution, was in the chair, and he was supported by Sir William Pope and other representatives of the two societies.

In the process continuous blowing-down of the boilers is combined with hot purification of the make-up feed water. The blow-down water is passed through a feed-water heater, the mud and separated salts are retained in a trap, and the purified blow-down water is returned to the boiler. When the feed-water, containing sodium carbonate and bicarbonate, gets into the boiler, the bicarbonate is decomposed and the carbonate partially hydrolysed, so that hydroxide, as well as carbonate, is used to purify the feed-water. The Kestner apparatus comprises a steam-separator, and a heater having a central reservoir to receive the blow-down liquid, and an annular vessel surrounding the reservoir, into which the purified blow-down water is decanted. The feed-water, pre-heated by the steam, mixes with the blow-down liquid as it leaves the reservoir, a measured amount of soda having been previously added, and precipitation and settlement take place in the annular chamber.

Dissolved oxygen is removed by means of cast iron containing 0.7 per cent. of manganese, which acts as a catalyst. The hydrated ferric oxide formed is reduced to the ferrous state on being brought in contact with the unchanged iron, and the ferrous compound is available for further de-gassing. The apparatus employed the water flows alternately through two vessels each initially filled with iron turnings, or it passes up and down a single vessel similarly charged and with filters at each end, the direction of flow being changed by a valve-arrangement, thus allowing the material to recuperate.



## NEWS FROM THE SECTIONS.

## GLASGOW.

A meeting of this Section was held in the Institute of Engineers and Shipbuilders, Glasgow, on February 1. Mr. J. H. Young occupied the chair, and Mr. J. G. Roberts read a paper on "Colloids in Clays: A Criticism."

The author gave a brief account of the better known colloids, typical suspensions, and emulsoids of the silicic acid type. He described the composition and character of china clay, and contrasted its "colloidal" properties and those of other purer clays with silicic acid and alumina gels. The contradictory nature of many of the statements in Searle's paper on "Colloids in Clay" contained in the third Report on Colloid Chemistry were pointed out and discussed, and also the apparent confusion between the colloid and amorphous conditions. The liquefaction of clay suspensions was demonstrated, and also electro-osmosis or cataphoresis of a suspension of fine clay. Fired samples of some electrically-separated clays were also shown.

## EDINBURGH AND EAST OF SCOTLAND.

At a meeting held on February 15 Dr. D. S. Jordan in the chair, a paper was read by Mr. W. N. Stokoe, entitled "Investigations on the Rancidity of Margarine and Butter Fats."

After referring to the chemical changes possibly occurring in rancid fats, the author discussed the chief causes of rancidity, which in commercial oils are the action of air, light and moisture, and lipolytic enzymes. Rancidity in butter and margarine is caused by the action of micro-organisms which find in the substances suitable media for their growth and propagation.

Mr. Stokoe classified the various forms of rancidity under the divisions of appearance, odour, and taste, and showed lantern slides of the micro-organisms associated with each form. A particular kind of rancidity, which he termed the "perfume" form, occurs only in those margarines containing coconut and palm-kernel oils, and is caused by enzymes secreted by such moulds as *Penicillium glaucum* and species of *Aspergillus*. The substance giving rise to the perfume consists chiefly of methylonyl ketone accompanied by other ketones. Other forms of rancidity were discussed and specimens of rancid fats exhibited.

The various sources of contamination by micro-organisms during the manufacture of butter and margarine were examined, and great emphasis was laid on the necessity for thorough cleanliness in manufacturing processes. The practice of transporting and storing oils and fats in wooden containers is utterly wrong, as it is impossible to sterilise the wood properly, and the canvas put on the bungs of barrels is a fruitful source of mould pores. The paper used for wrapping margarine also gives rise to contamination by micro-organisms, and the author described various methods of sterilising wrapping paper.

## NEWCASTLE.

Two papers were read at the meeting held in Armstrong College on February 16, Dr. J. H. Atkinson presiding, both of which described work carried out in the Agricultural Chemistry Laboratory of the College.

Dr. A. A. Hall described "A Quick Volumetric Method for the Estimation of Mercury," which is applicable to soluble mercuric salts either in the presence of acid or of any substance unaffected by caustic soda solution, and can also be used in the presence of soluble copper salts and ammonium salts. An excess of an ammonium salt and then an

excess of the standard alkali is added to a known volume of the mercuric solution, and the "white precipitate" formed is made up to a known volume with water and filtered through a dry filter. An aliquot part of the filtrate is titrated with standard acid in presence of methyl orange, and the difference between the amounts of alkali and acid used represents the amount of alkali consumed in precipitating the mercury. If acid be present it can be estimated by titration with alkali before the excess of alkali is added. If the solution contains copper the deep-blue solution formed gradually fades as the liquid becomes neutralised. The author adduced analytical results which indicated the accuracy of the method under the various conditions mentioned.

In a paper on "Fractional Distillation Columns," Mr. S. Hoare Collins described a short fractionating column about 30 cm. long from bottom to side tube and 18 cm. diameter with an extension tube 90 cm. long of the same diameter. Both are filled with glass spirals made by winding heated glass rod of 5 mm. diameter round a central rod of 6 cm. diameter. Experiments conducted with a mixture of benzene and toluene showed that such a spiral gave far better results than a "rod and disc" filling.

## MANCHESTER.

The sixth meeting of the session was held at the Textile Institute on March 4, Mr. Allan presiding. About fifty members were present and two papers were read. The first, on "Column Apparatus for the Chemical Industry," read by Mr. S. J. Tungay, set out the conditions to be observed in the design and construction of column apparatus, and emphasised the point that faulty working was in most cases due to disregard of the fundamental actions required to take place in the columns; as an instance it was mentioned that counter currents may mix or may not. The paper contained descriptions of many types of column apparatus. The second paper, on "Normal Amyl Benzene and some of its Derivatives," by Messrs. L. G. Radcliffe and N. Simpkin, was read by the former, and described investigations made to find uses for butyl alcohol now being produced in large quantity. The production of a mono-sulphonic-acid phenol and a para nitro-compound was described.

## MEETINGS OF OTHER SOCIETIES.

## THE INSTITUTE OF CHEMISTRY.

The forty-third annual general meeting was held on March 1 at Russell Square, W.C. Sir Herbert Jackson, the retiring president, in his address remarked that Government departments and official authorities generally had of late extended greater recognition to the services of men of science than in the past. The conditions of appointment of chemists, both in the Government service and in industry, had been improved, and until the close of 1920 very few members of the Institute were disengaged. The prevailing depression in industry had, however, affected the employment of chemists, but even now less than 50 were disengaged out of nearly 3300 on the roll. The by-laws of the Institute had been revised to provide for the representation of chemists from various parts of the country and practising in different departments of work, to define more specifically certain points relating to the ethics of the profession, and to restrict the membership of the Institute to British subjects. The Institute was taking part in many matters affecting the public life of the country where chemistry was concerned, and the annual report showed that chartered professional bodies of this

character were able to render valuable service to the State. It was probably desirable at the present moment for the Council of the Institute, without taking part in politics, to express its views on the grave importance of maintaining in this country industries on which not only the future development of our chemical industry and many allied industries depends, but the future of a very large number of students of chemistry who are now being trained. The Institute was entrusted by its charter to secure the supply of well-trained chemists, but unless a great chemical industry was maintained their prospects would be very poor. The president maintained that the Institute had throughout its existence fulfilled the purposes for which it was incorporated. It had kept alive the corporate spirit of the profession; it was the officially recognised public body to which the country could turn for advice and guidance on matters relating to the profession; it assisted those who intended to follow the profession of chemistry, and promoted and maintained the highest standard of training and competence for that profession; it registered the trained and competent, and it fostered the strictest integrity on the part of its members in their dealings with one another and with the public. He was confident that it would continue to gain strength and prestige.

#### SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting was held at the Chemical Society's Rooms on March 2, Mr. A. Smetham presiding.

A paper on "The Acidity of Ink and the Action of Bottle Glass on Ink" was read by Mr. C. A. Mitchell, who explained why acid was added to ink, gave the amount present in standard inks, and described acid-free inks, the estimation of acidity, and the difficulty caused by the presence of dye-stuffs. The uses and limitations of the hydrogen-peroxide method and the estimation of total acidity as a check on the manufacturing process were dealt with and the effects of glass on ink discussed.

In a paper on "The Detection of Adulteration in Butter by means of the Melting-point of the Insoluble Volatile Acids," Mr. G. van B. Gilmour described how the addition of small amounts of coconut and palm-kernel fats to butter appreciably lowers the melting-point of the insoluble volatile acids. Tables of melting-points and distillation-figures for pure fats and mixtures were given, and also graphs showing how the melting-point is affected by adding increasing quantities of coconut and of palm-kernel fat.

Messrs. S. H. Blichfeldt and T. Thornley described a method and apparatus for routine determinations of melting-points of fat and fatty acids, in which a column of melted fat 1 cm. long in a tube of 1 mm. bore, 3 mm. diameter, and 60 mm. length, is rapidly solidified on ice and kept there for two hours; the tube is then placed in a water-bath with the upper level of the column 1 cm. below the surface, and heated at the rate of 1° C. per minute. The temperature at which the column rises is taken as the melting-point. The apparatus consists of a rectangular glass water-bath holding twenty tubes, which is heated, stirred, and illuminated electrically.

#### THE CHEMICAL SOCIETY.

At the meeting held on March 3, Sir James Dobbie presiding, the following papers were read in abstract:—(1) "The Chemistry of the Glutaconic Acids. Pt. XII. The simultaneous occurrence of 1:2- and of 1:3-addition to glutaconic ester. A study in mobile equilibrium involving the utilisation of the labile ester in the 'nascent' condition". C. K. Ingold and J. F. Thorpe. (2) "A Second

Form of 8:6'-Dinitrodiphenic Acid and its Conversion into New Cyclic Systems": J. Kenner and W. V. Stubbings. (3) "The Structural Isomerism of Oximes. Pt. III. A fourth benzildioxime": F. W. Atack and L. Whioyates.

The president announced that the guests of honour who will be entertained at the Anniversary Dinner to be held on March 17 are: Sir James Dewar, Sir Edward Thorpe, and Sir William Tilden, all past-presidents who have attained their jubilee as Fellows of the Society.

### CORRESPONDENCE.

#### ORIGIN OF THE SOCIETY OF CHEMICAL INDUSTRY.

SIR,—It is difficult to understand the misleading statements made by Mr. Douglas Hermann in his letter relating to the inception of the Society of Chemical Industry which appeared in the *Journal* of January 31; and, as one concerned in the formation of this Society, I feel constrained to place before your readers the following facts relating thereto:—

On the occasion of a visit to Newcastle, Mr. T. W. Stuart (then manager of Messrs. Tennant's works, and now the United Alkali Co.'s general manager) invited me to attend a meeting of what was then known as The Tyne Chemical Society. Realising the great advantages accruing from the intimate association and frequent conferences of those engaged in chemical manufacture, on my return to Widoes I convened a meeting of chemists and managers of this district, which was held in the Y.M.C.A. rooms of this town, on November 21, 1879, to consider the advisability of forming a local chemical society similar to the one referred to. The following gentlemen attended that meeting: Messrs. MacIllwain, Northing, Tallon, Williamson, Taylor, Marsh, Lord, Campbell, Bottomley, Coxon, and Hargreaves. Messrs. MacIllwain, Tallon, Coxon, Campbell, with myself as hon. sec., were appointed an executive committee and held a meeting on December 1, 1879, when it was resolved that a general meeting be held on December 5, at which the following recommendation, among others, should be presented for consideration: "That the name of the society be the South Lancashire Chemical Society."

When sending out the usual notices for this general meeting, I wrote Mr. James Muspratt, inviting him to be present. The meeting was held in due course and was presided over by Mr. James Muspratt (who had not hitherto attended the preliminary meetings). It was resolved that a special committee be formed to draft rules to be placed before the next general meeting, and also to arrange time and place for such meeting. This was held on January 29, 1880, at the Royal Institution, Liverpool, Dr. Mond presiding. The following letter was received from Prof. Roscoe in reply to the secretary's invitation asking him to attend that meeting:—

"The Owens College, Manchester,

Jan. 24, 1880.

"DEAR SIR,—In reply to your circular of yesterday's date, I beg to say that I consider your proposal to found a Chemical Society for Lancashire on the basis of the Newcastle Society a very useful one, and it will give me much pleasure to assist those interested in its formation in every way in my power. I regret, however, that my engagements next week prevent me from attending the meeting in Liverpool on Thursday.—Yours truly,

"H. E. Roscoe.

"John Hargreaves, Esq."

At a further meeting, held at Owens College, on April 19, 1880, the late Mr. G. E. Davis (whom I had approached privately), was appointed hon. secretary. During that meeting a few gentlemen, at Mr. Davis's suggestion, withdrew to an adjoining room, where our first subscriptions to the Society were paid. Up to that time all the expenses had been borne by me personally, and Mr. Davis insisted that I be reimbursed from the monies he then held as subscriptions. Concerning the Faraday Club and the alleged jealousy of the Widnes chemists, referred to by Mr. Hermann, I may say that the existence of this club only became known to me at that time through a local chemist when he was invited to join the projected Society.

Asked by Mr. Davis some years after the formation of the Society whether I knew the reason he attended the meeting held in the Drill Hall on December 5, 1879, over which Mr. James Muspratt presided, he declared, "I came to smash it up." Fortunately for the Society, "coming to scoff, he remained to pray." I know of no man who, collaborating with the late Mr. Tyrer, would or could have so efficiently devoted the very large amount of time and energy required in mothering the infant Society and establishing it on its present firm foundation, and without whom the Society could never have become such an influential organisation.

I give the above details—which are only a few of the facts I have available—as there seems to be so much misconception still prevalent concerning the origin of the Society of Chemical Industry.—I am, Sir, etc., JOHN HARGREAVES.

Widnes, March 1, 1921.

## PERSONALIA.

The President of the Society, Sir William J. Pope, has been elected "Membre d'Honneur" of the Société de Chimie Française.

Mr. A. Chaston Chapman has been elected president of the Institute of Chemistry, in succession to Sir Herbert Jackson.

The Perkin Medal of the Society of Dyers and Colourists has been awarded to Mr. H. A. Lowe for his invention of the process for mercerising cotton under tension.

Mr. A. J. Greenaway has been appointed editor of the Journal of the Chemical Society, in succession to Dr. J. C. Cain, and Dr. Clarence Smith becomes assistant editor.

The fifteen candidates who have been selected by the Council of the Royal Society to be recommended for the Fellowship include Dr. F. W. Aston, Prof. V. L. Bragg, Prof. K. J. P. Orton, and Prof. J. C. Philip.

Dr. M. O. Forster will preside over Section B (Chemistry) of the British Association for the Advancement of Science at the meeting to be held from September 7 to 14, in Edinburgh. Sir Edward Thorpe is president-elect of the Association.

Prof. J. v. Braun, of the Berlin Agricultural Hochschule, has accepted the chair of chemistry at the University of Frankfurt as successor to Prof. Freund, and Prof. H. Fühner, of Königsberg, has been called to the chair of pharmacology in the University of Leipzig, in succession to Prof. R. Boehm, retired.

The testing station for new medicinal preparations recently established at Berlin has been placed under the direction of Geheimrat Heffter, of the Pharmacological Institute, Berlin University, and Prof. Holste, in Jena, has been given charge of the headquarters of the Medicinal Preparations Commission of the Society for Internal Medicine.

With deep regret we record the death of the Rt. Hon. Lord Moulton, on March 9 at the age of 75.

Sir Charles A. Cameron, superintendent medical officer of public health and city analyst to the Dublin Corporation, died on February 27, at the age of 90.

The death is announced of Dr. R. Suchsland, who for over fifty years was chief chemist to Messrs. Vivian and Sons, of Swansea. The deceased was born in Frankfurt in 1839, was engaged by the late Lord Swansea in 1861, and became naturalised in the early seventies.

The death of Dr. Ignaz Stroof, one of the pioneers of the German heavy chemical industry, took place in November last, in his eighty-third year. His most important work was the introduction of the electrolytic method of manufacturing caustic alkali by the diaphragm process.

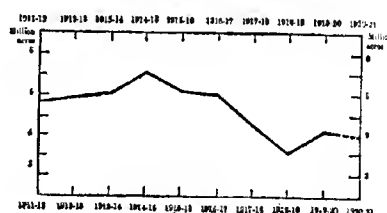
We regret to record the death in February of Mr. John R. Griffin, of Messrs. J. J. Griffin and Co., Ltd., who joined the Society in 1890, and recently took a very active part in the work of the committee appointed by the Council to investigate the standardisation of laboratory glassware (*cf.* J., Aug. 15, 1919; also the death of Mr. S. H. Blichfeldt on March 3, after a very short illness).

## NEWS AND NOTES.

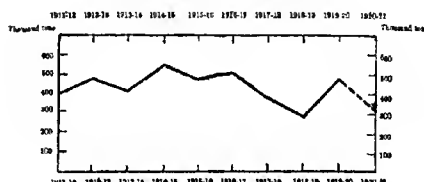
### BRITISH INDIA.

**The Sesamum Industry.**—The *Indian Trade Journal* of January 28 contains the detailed final memorandum on the sesamum crop of 1920-21, together with charts (reproduced below) showing the present estimates of the total area and yield as compared with the final figures of the preceding nine years:—

Area under Sesamum (in million acres).



Yield of Sesamum (in thousand tons).



Exclusive of Burma, which contains nearly 25 per cent. of the area under sesamum in British India, the total area under the crop is reported to be 3,964,000 acres, or 2 per cent. below the corresponding revised estimate last year, and the total yield (Hyderabad and Burma excluded) is estimated at 323,000 tons, which is 20 per cent. below the forecast this time last year. The condition of the crop is reported to be fair on the whole.

character were able to render valuable service to the State. It was probably desirable at the present moment for the Council of the Institute, without taking part in politics, to express its views on the grave importance of maintaining in this country industries on which not only the future development of our chemical industry and many allied industries depends, but the future of a very large number of students of chemistry who are now being trained. The Institute was entrusted by its charter to secure the supply of well-trained chemists, but unless a great chemical industry was maintained their prospects would be very poor. The president maintained that the Institute had throughout its existence fulfilled the purposes for which it was incorporated. It had kept alive the corporate spirit of the profession; it was the officially recognised public body to which the country could turn for advice and guidance on matters relating to the profession; it assisted those who intended to follow the profession of chemistry, and promoted and maintained the highest standard of training and competence for that profession; it registered the trained and competent, and it fostered the strictest integrity on the part of its members in their dealings with one another and with the public. He was confident that it would continue to gain strength and prestige.

#### SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting was held at the Chemical Society's Rooms on March 2, Mr. A. Smetham presiding.

A paper on "The Acidity of Ink and the Action of Bottle Glass on Ink" was read by Mr. C. A. Mitchell, who explained why acid was added to ink, gave the amount present in standard inks, and described acid-free inks, the estimation of acidity, and the difficulty caused by the presence of dye-stuffs. The uses and limitations of the hydrogen-peroxide method and the estimation of total acidity as a check on the manufacturing process were dealt with and the effects of glass on ink discussed.

In a paper on "The Detection of Adulteration in Butter by means of the Melting-point of the Insoluble Volatile Acids," Mr. G. van B. Gilmour described how the addition of small amounts of coconut and palm-kernel fats to butter appreciably lowers the melting-point of the insoluble volatile acids. Tables of melting-points and distillation-figures for pure fats and mixtures were given, and also graphs showing how the melting-point is affected by adding increasing quantities of coconut and of palm-kernel fat.

Messrs. S. H. Blichfeldt and T. Thornley described a method and apparatus for routine determinations of melting-points of fat and fatty acids, in which a column of melted fat 1 cm. long in a tube of 1 mm. bore, 3 mm. diameter, and 60 mm. length, is rapidly solidified on ice and kept there for two hours; the tube is then placed in a water-bath with the upper level of the column 1 cm. below the surface, and heated at the rate of 1° C. per minute. The temperature at which the column rises is taken as the melting-point. The apparatus consists of a rectangular glass water-bath holding twenty tubes, which is heated, stirred, and illuminated electrically.

#### THE CHEMICAL SOCIETY.

At the meeting held on March 3, Sir James Dohbie presiding, the following papers were read in abstract:—(1) "The Chemistry of the Glutaconic Acids. Pt. XII. The simultaneous occurrence of 1:2- and of 1:3-addition to glutaconic ester. A study in mobile equilibrium involving the utilisation of the labile ester in the 'nascent' condition": C. K. Ingold and J. F. Thorpe. (2) "A Second

Form of 6:6'-Dinitrodiphenic Acid and its Conversion into New Cyclic Systems": J. Kenner and W. V. Stubblings. (3) "The Structural Isomerism of Oximes. Pt. III. A fourth benzildioxime": F. W. Attack and L. Whinyates.

The president announced that the guests of honour who will be entertained at the Anniversary Dinner to be held on March 17 are: Sir James Dewar, Sir Edward Thorpe, and Sir William Tilden, all past-presidents who have attained their jubilee as Fellows of the Society.

### CORRESPONDENCE.

#### ORIGIN OF THE SOCIETY OF CHEMICAL INDUSTRY.

SIR,—It is difficult to understand the misleading statements made by Mr. Douglas Hermann in his letter relating to the inception of the Society of Chemical Industry which appeared in the *Journal* of January 31; and, as one concerned in the formation of this Society, I feel constrained to place before your readers the following facts relating thereto:—

On the occasion of a visit to Newcastle, Mr. T. W. Stuart (then manager of Messrs. Tennant's works, and now the United Alkali Co.'s general manager) invited me to attend a meeting of what was then known as The Tyne Chemical Society. Realising the great advantages accruing from the intimate association and frequent conferences of those engaged in chemical manufacture, on my return to Widnes I convened a meeting of chemists and managers of this district, which was held in the Y.M.C.A. rooms of this town, on November 21, 1879, to consider the advisability of forming a local chemical society similar to the one referred to. The following gentlemen attended that meeting: Messrs. MacIllwain, Northing, Tallon, Williamson, Taylor, Marsh, Lord, Campbell, Bottomley, Coxon, and Hargreaves. Messrs. MacIllwain, Tallon, Coxon, Campbell, with myself as hon. sec., were appointed an executive committee and held a meeting on December 1, 1879, when it was resolved that a general meeting be held on December 5, at which the following recommendation, among others, should be presented for consideration: "That the name of the society be the South Lancashire Chemical Society."

When sending out the usual notices for this general meeting, I wrote Mr. James Muspratt, inviting him to be present. The meeting was held in due course and was presided over by Mr. James Muspratt (who had not hitherto attended the preliminary meetings). It was resolved that a special committee be formed to draft rules to be placed before the next general meeting, and also to arrange time and place for such meeting. This was held on January 29, 1880, at the Royal Institution, Liverpool, Dr. Mond presiding. The following letter was received from Prof. Roscoe in reply to the secretary's invitation asking him to attend that meeting:—

"The Owens College, Manchester,

Jan. 24, 1880.

"DEAR SIR,—In reply to your circular of yesterday's date, I beg to say that I consider your proposal to found a Chemical Society for Lancashire on the basis of the Newcastle Society a very useful one, and it will give me much pleasure to assist those interested in its formation in every way in my power. I regret, however, that my engagements next week prevent me from attending the meeting in Liverpool on Thursday.—Yours truly,

"H. E. Roscoe.

"John Hargreaves, Esq."

At a further meeting, held at Owens College, on April 19, 1880, the late Mr. G. E. Davis (whom I had approached privately), was appointed hon. secretary. During that meeting a few gentlemen, at Mr. Davis's suggestion, withdrew to an adjoining room, where our first subscriptions to the Society were paid. Up to that time all the expenses had been borne by me personally, and Mr. Davis insisted that I be reimbursed from the monies he then held as subscriptions. Concerning the Faraday Club and the alleged jealousy of the Widnes chemists, referred to by Mr. Hermann, I may say that the existence of this club only became known to me at that time through a local chemist when he was invited to join the projected Society.

Asked by Mr. Davis some years after the formation of the Society whether I knew the reason he attended the meeting held in the Drill Hall on December 5, 1879, over which Mr. James Muspratt presided, he declared, "I came to smash it up." Fortunately for the Society, "coming to scoff, he remained to pray." I know of no man who, collaborating with the late Mr. Tyrer, would or could have so efficiently devoted the very large amount of time and energy required in mothering the infant Society and establishing it on its present firm foundation, and without whom the Society could never have become such an influential organisation.

I give the above details—which are only a few of the facts I have available—as there seems to be so much misconception still prevalent concerning the origin of the Society of Chemical Industry.—I am, Sir, etc.,

JOHN HARGREAVES.

Widnes, March 1, 1921.

## PERSONALIA.

The President of the Society, Sir William J. Pope, has been elected "Membre d'Honneur" of the Société de Chimie Française.

Mr. A. Chaston Chapman has been elected president of the Institute of Chemistry, in succession to Sir Herbert Jackson.

The Perkin Medal of the Society of Dyers and Colourists has been awarded to Mr. H. A. Lowe for his invention of the process for mercerising cotton under tension.

Mr. A. J. Greenaway has been appointed editor of the Journal of the Chemical Society, in succession to Dr. J. C. Cain, and Dr. Clarence Smith becomes assistant editor.

The fifteen candidates who have been selected by the Council of the Royal Society to be recommended for the Fellowship include Dr. F. W. Aston, Prof. W. L. Bragg, Prof. K. J. P. Orton, and Prof. J. C. Philip.

Dr. M. O. Forster will preside over Section B (Chemistry) of the British Association for the Advancement of Science at the meeting to be held from September 7 to 14, in Edinburgh. Sir Edward Thorpe is president-elect of the Association.

Prof. J. v. Braun, of the Berlin Agricultural "Hochschule," has accepted the chair of chemistry at the University of Frankfurt as successor to Prof. Freund, and Prof. H. Fühner, of Königsberg, has been called to the chair of pharmacology in the University of Leipzig, in succession to Prof. R. Boehm, retired.

The testing station for new medicinal preparations recently established at Berlin has been placed under the direction of Geheimrat Heffter, of the Pharmacological Institute, Berlin University, and Prof. Holste, in Jena, has been given charge of the headquarters of the Medicinal Preparations Commission of the Society for Internal Medicine.

With deep regret we record the death of the Rt. Hon. Lord Moulton, on March 9 at the age of 75.

Sir Charles A. Cameron, superintendent medical officer of public health and city analyst to the Dublin Corporation, died on February 27, at the age of 90.

The death is announced of Dr. R. Suchsland, who for over fifty years was chief chemist to Messrs. Vivian and Sons, of Swansea. The deceased was born in Frankfurt in 1839, was engaged by the late Lord Swansea in 1861, and became naturalised in the early seventies.

The death of Dr. Ignaz Stroof, one of the pioneers of the German heavy chemical industry, took place in November last, in his eighty-third year. His most important work was the introduction of the electrolytic method of manufacturing caustic alkali by the diaphragm process.

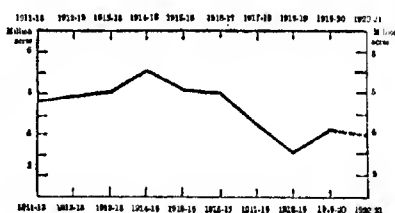
We regret to record the death in February of Mr. John R. Griffin, of Messrs. J. J. Griffin and Co., Ltd., who joined the Society in 1890, and recently took a very active part in the work of the committee appointed by the Council to investigate the standardisation of laboratory glassware (*cf. J.*, Aug. 15, 1919; also the death of Mr. S. H. Blichfeldt on March 3, after a very short illness).

## NEWS AND NOTES.

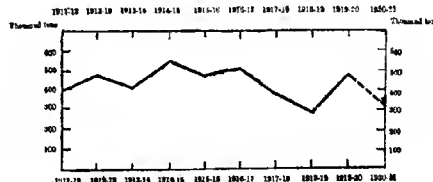
### BRITISH INDIA.

**The Sesamum Industry.**—The *Indian Trade Journal* of January 28 contains the detailed final memorandum on the sesamum crop of 1920-21, together with charts (reproduced below) showing the present estimates of the total area and yield as compared with the final figures of the preceding nine years:—

Area under Sesamum (in million acres).



Yield of Sesamum (in thousand tons).



Exclusive of Burma, which contains nearly 25 per cent. of the area under sesamum in British India, the total area under the crop is reported to be 3,964,000 acres, or 2 per cent. below the corresponding revised estimate last year, and the total yield (Hyderabad and Burma excluded) is estimated at 323,000 tons, which is 20 per cent. below the forecast this time last year. The condition of the crop is reported to be fair on the whole.

**Indigo Cultivation in Assam.**—Recent experiments appear to indicate that indigo grown in Assam will prove a formidable competitor of synthetic indigo. Plants have been grown in Assam containing 0.7 per cent. of indigotin in the leaf, yielding 60–80 lb. of cake indigo per acre. A company called "Assam Indigo, Ltd.," has been formed for the purpose of producing indigo by modern methods.—(*U.S. Com. Rep.*, Feb. 4, 1921.)

#### AUSTRALIA.

**Oil Exploration.**—Stimulated by the Government's offer of a prize of £50,000, the search for oil in paying quantities is being pursued vigorously. The Northern Territory, Papua, and South Australia are being explored, but so far only the Roma bore in Queensland and the bores in Papua are considered hopeful; in neither of these localities has oil yet been produced on a commercial scale.

#### New South Wales.

**Cultivation of Groundnuts.**—Many farmers are now cultivating this crop. The heavy loams of the northern coast of the State are the most suitable, but some good crops have been obtained on the Murrumbidgee irrigation areas. A firm in Sydney is erecting an oil-extraction plant which, it is hoped, will be in full working order before the next crop is harvested.

#### Victoria.

**Chemistry at Melbourne University.**—In commemoration of its late chairman, Mr. James Cumming, a leader of Victorian chemical industry, the directors of Cumming, Smith and Co., Pty., Ltd., have offered the sum of £5000 towards the erection and equipment of a research laboratory and lecture-theatre in the University of Melbourne. The offer is being considered by the council of the University.

#### Western Australia.

**Forest Products.**—The following notes have been abstracted from the recently issued Report of the Conservator of Forests for the year 1919–20. The gross value of exported timber during the year, including sandalwood, amounted to £743,373, an increase of £263,307 over the previous year.

**Kiln Drying.**—The future of kiln drying of jarrah being assured, both by the results obtained by the Department and by local firms in their commercial driers, the timber kiln erected at Crawley has been operated during the year with a view to overcoming some of the difficulties encountered. It is hoped that before long all the information required to enable such kilns to be operated easily and successfully will be available.

**Tan Barks.**—The value of mallet bark exported amounted to £22,121, or £3246 more than in 1918–19. Mangrove bark has come into prominence, analysis having shown that the black mangrove (*Bruguiera gymnorhiza*) contains 46 per cent. and the red mangrove (*Ceriops candolleana*) 44 per cent. of tannin. Marri kino tannage continues to be used for sole and harness leather. The tanneries of Western Australia are still dependent on Natal wattle bark, and every attempt should be made to find local substitutes to reduce this importation. The trees and shrubs in the State contain sufficient tanning-bearing materials not only for local tanneries, but also for export.

**Sandalwood.**—This trade is a purely Chinese one, and the average quantity of sandalwood absorbed by China during the past 70 years is 4500 tons per annum. During the year under review, however, the cnt amounted to 17,269 tons. This very large increase is difficult to explain, but the main cause is probably the very high rate of exchange which followed the rise in the price of silver, and consequently of the Chinese dollar. Owing to the alarming rate of cutting, an attempt was made to

regulate the industry by raising the royalty on the wood from 5s. to £2 per ton. Although this gives a considerably increased revenue, it does not affect the main object of regulating the supply of wood to that quantity which China can absorb, and so assuring a continuous trade at a maximum price. During the year 5572 lb. of sandalwood oil was extracted locally, an increase of 1852 lb. over the previous year's output.

**Kingia Grass Tree Fibre Industry.**—This new industry is developing gradually and the excellence of the fibre for broom making and other purposes has been proved. During the year the "Dibinya" fibre works shipped 27 tons to the Eastern States, and a second factory has just started operations. So far only the fibre of this tree has been used; the outside is stripped in the bush and the inside core also goes to waste. This is a matter demanding immediate attention, for at the present time only about one-fifth of the crop is being utilised.

#### CANADA.

**Glass Production in Vancouver.**—A large plant for the manufacture of table ware is under construction at Westmoreland, near Huntingdon, in West Vancouver, for the West Virginia Glass Manufacturing Co.; it will include a glass furnace, said to be the largest in the world, employ 400–500 work-people, and be ready for a trial run in April.—(*Oil, Paint and Drug Rep.*, Jan. 31, 1921.)

#### FRANCE.

**Industrial Notes.**—**Coal.**—The French coal industry was decontrolled on March 1, and the supertax of 28 per cent. abolished; the pithead tax is retained in order to keep down prices. The supertax was intended to equalise the prices of home and foreign coal, but as the differences in price have of late been reduced by nearly 50 per cent. its continuance is unnecessary. The total output of coal in France, including Lorraine, was 25,276,304 tons in 1920, compared with 22,476,766 t. in 1919. The total production of coke made from Saar coal is about 87,000 t. monthly; the average selling price of the coal and coke is about 97 francs per ton, and the profit 13 fr. per ton, half of which has to be applied to extension work. In view of the possible eventual restoration of the Saar mines to Germany, it is proposed to exploit them to the fullest extent.

**Metallurgy.**—Opposition to the great metallurgical syndicates continues to grow. The Comptoir de Longwy, as already announced, has been liquidated; it is rumoured that the Comptoir des Fontes Hematites will soon share the same fate; and the Comptoir Sidérurgique, which sells and distributes semi-manufactured and manufactured products, is also becoming unpopular. The opposition emanates from the federation of producers that has been formed since the war, and which by reason of its control of blast furnaces, steel works, rolling mills and a powerful selling organisation, no longer requires intermediaries such as the syndicates mentioned above. It is, however, doubtful if these can be permanently dispensed with, as the country is already producing twice as much pig iron as it consumes, and central organisations will be required to dispose of the surplus production to foreign buyers.

**Chemical Industry.**—German competition in fine chemicals is steadily growing. French producers who had found a market in Brazil, Chile, and Argentina are being gradually ousted by Darmstadt and Hamburg firms which undersell them by about 10 per cent. on quoted prices. Competition is not so active in heavy chemicals on account of the long periods required for delivery and of the high transport rates. The prohibition of the export of new and scrap celluloid, oil of turpentine and fertilisers has been revoked in order to facilitate the liquidation of existing stocks.



## UNITED STATES.

**Treatment of Alkaline Soils with Sulphuric Acid.**—Considerable interest has been aroused by favourable results obtained in the south-west by the direct application of sulphuric acid to adobe soils. Plots upon which nothing had been raised hitherto proved to be fertile after this treatment. This is said to be due both to the correction of super-alkalinity and to improvement in the physical character of soils treated.

**Extraction of Potassium Chloride and Borax from Natural Brines.**—At Keeler, California, a new process is being worked for recovering potassium chloride and borax in marketable form from natural brines. After securing the greatest possible concentration by solar evaporation the liquor is treated with carbon dioxide to free it from the carbonates of soda. It is then concentrated further in double-effect evaporators and submitted to fractional crystallisation.

**Propyl Alcohol from Wood Waste.**—A process for making propyl alcohol from the waste gases of the Burton petroleum-cracking still is being worked commercially. These gases contain large quantities of propylene, and after hydrogen sulphide has been removed they are passed through sulphuric acid, which combines with the propylene, and propyl alcohol is then obtained by distillation. Iso-propyl alcohol can be oxidised directly to acetone, or a series of esters of commercial importance can be made from it. The new alcohol will be called "petrohol," and the present capacity of the plant is 250 galls. per day. It is anticipated that this alcohol will be produced in competition with methanol, the new commercial term for wood alcohol, as a solvent for gums and resins.

**The Chemical Warfare Service.**—The American Chemical Society, through its Committee on National Policies, has communicated the following resolution to members of the Senate and the House of Representatives:—

"While in complete accord with the spirit prompting the restrictions of appropriations by the present Congress, nevertheless the American Chemical Society's Committee on National Policies would urge upon the Congress more favourable provision for the Chemical Warfare Service than is contemplated by the amount set by the House of Representatives—\$1,500,000. The carefully prepared estimates of the officers of that Service, slightly less than \$4,500,000, represent less than 1½ per cent. of the total appropriation for the army carried in the House bill. This amount is to care for the valuable property of the Government at Edgewood Arsenal, to enable the continuation of research on new lines of defence and offence, and to provide for the training of special troops and for the instruction of the entire army in all features of gas warfare. In view of the tremendous increase in the use of gases during the last year of the war, and of the fact that approximately 30 per cent. of the casualties of our army in the war was due to gas wounds, we feel that the proposed reduction to one-third of the appropriation asked would so seriously cripple the development of the Chemical Warfare Service as to constitute a matter of grave national concern. We therefore urge that the Congress appropriate the original amount asked for the Service in the estimates submitted."

**Mica in 1918.**—A report of the United States Geological Survey issued in 1920 gives the production of mica in that country in 1918 as 3114 short tons, which comprised 822 t. of sheet and 2292 t. of scrap mica. As in previous years (*cf.* J., 1918, 437 r), the greatest production was in North Carolina, but increasing quantities of excellent mineral are now being obtained from the Thomaston field in Georgia. Production is apparently at its maxi-

mum, the stimulus of high prices and a ready market having had little effect. Of the mica imported in 1918, 54 per cent. came from India, 26 per cent. from Canada, and 15 per cent. from Brazil. The value of the imports, \$1,541,129, was approximately double that of the exports.

No figures are available for the world's production in 1918, but India and Canada produced 3062 and 374 t. respectively. To-day the largest consumers are the United States and Great Britain, followed by France and Italy, Germany having lost her leading position in the European electrical industry to England. The future development of mica deposits depends to some extent on the success of various substitutes, *e.g.*, compressed-paper products, bakelite, etc. So far there appears to be no satisfactory substitute for sheet mica used in electrical insulation, for which purpose no less than 86 per cent. of the sheet mica and splittings produced is utilised. About 98 per cent. of the world's output of sheet mica is supplied by India and the United States (muscovite) and Canada (phlogopite). The demand keeps pace with increasing production, and important developments may be expected in British East Africa, Nyassaland, and Rhodesia.

**Nitrobenzene-Poisoning.**—A peculiar case of poisoning due to nitrobenzene has been reported. A workman who treated his shoes with a preparation containing this substance and wore them immediately was taken ill within three hours. The desirability of not wearing shoes so treated within three or four days of treatment and applying the substance in a well-ventilated place are emphasised.

## SOUTH AFRICA.

**Industrial Progress.**—The report of the General Manager of Railways and Harbours for the year 1919-20 records considerable industrial expansion. Although attention has been devoted mainly to expanding and modernising existing industries, new industries are still springing up, and they have been to a great extent shielded from foreign competition by the continuance of war-prices in exporting countries. Compared with the year 1915-16, there are 2044 additional factories, 41,910 more employees, and the value of the production has increased by £30,000,000.

The iron and steel industry should in time become one of the most important in the Union. Although small it is making satisfactory progress. In 1919 the production of iron and steel goods from local furnaces was valued at £406,000, and 1967 tons of pig iron, valued at £12,500, was produced from local ore. The trade in wattle bark and wattle-bark extract continues to grow; 96,124 tons of bark was shipped from Durban during the year ended March, 1920, and eleven mills for making extract have been established or are in course of erection. The value of the exports in this industry has increased from £195,000 in 1909 to £603,000 in 1919. The tanning and leather goods industry has been active throughout the Union, notably at Durban, Port Elizabeth, King William's Town, Wellington, Pretoria, and Middleburg. There is a ready market for local products, and the factories are unable to keep pace with the demand. Leather and leather goods to the value of £62,000 were exported in 1919, or £56,000 more than in 1909.

## JAPAN.

**The Cement Industry.**—The Nagoya Cement Co. has recently completed its plant at Tokyo, and the output, which it is anticipated will reach 200,000 barrels per annum, will be taken largely by hydro-electric companies. The company owns mining rights for limestone in the Shiga and Miye Prefectures.—(*U.S. Com. Rep.*, Jan. 4, 1920.)



## GENERAL.

**Mercerised Cotton.**—As announced on page 872, the Perkin Medal of the Society of Dyers and Colourists has been awarded to H. A. Lowe for his invention of mercerised cotton under tension.

The process of treating cotton with a strong solution of caustic soda produces a change in the fibre known as "mercerisation," a word derived from the name of John Mercer, who first drew attention to the phenomenon in 1844, but today it is more particularly associated with cotton upon which a lustre has been produced by treatment with caustic soda while under tension. The production of a permanent lustre was the discovery of Horace A. Lowe, whose patents were taken out in 1889 and 1890. Lowe, then a young chemist in a Lancashire bleach-works, experienced so much difficulty in getting the process taken up that he allowed his patents to lapse. The process was first carried out on a commercial scale in 1895 by Thomas and Prevost of Crefeld, who took out a patent, apparently in ignorance of Lowe's previous discovery. The mercerised yarns produced by this firm met with such success that the process was widely taken up. In the litigation which naturally followed, the priority of Lowe's invention was clearly established, and Thomas and Prevost's patent was annulled. Lowe subsequently carried out his process at a works opened for the purpose near Manchester, and after a short time this was taken over by the Fine Cotton Spinners' Association, who began the production of mercerised yarns on a very large scale. Today mercerisation is one of the most important processes in use in the cotton dyeing and finishing trade.

Former recipients of the Perkin Medal have been von Baeyer for the synthesis of Indigo, Graebe and Liebermann for the synthesis of Alizarin, de Chardonnet for the invention of the first artificial silk, and Prof. Arthur Green for the discovery of Primuline.

"**Bismuth (1913-1919)**" (pp. 14, price 6d.) has been recently published by the Imperial Mineral Resources Bureau as an advance "part" of its annual volume on the Mineral Resources of the British Empire and Foreign Countries. The Bureau does not appear to be able to add appreciably to our knowledge concerning the production and marketing of bismuth ores, metal, or compounds. The table headed "World's Production of Bismuth" shows figures which are of little value, as it only gives the production of ore, concentrates, residues, etc., without stating the bismuth content and without giving the values. The largest producing country is Bolivia, which in 1918 was stated to have produced 245 tons of bismuth ingots, presumably sent to England to be refined. As a rule, the Bolivian native bismuth and high-grade concentrates are shipped to Europe and the United States for treatment, only the low-grade concentrates carrying less than 10 per cent. bismuth being smelted locally. Australia is a producer on a considerable scale and, although no figures are given for the production in the United States, the supply obtained during the process of smelting and refining lead and copper ores in that country must be an important source of the metal. Smaller quantities of bismuth ore have been produced in Rhodesia, India, Burma, China, and Norway. Figures for Germany are not available, but this country is normally an important producer.

During the war there was an increased demand for bismuth both for the manufacture of certain low-temperature fuses and for medicinal purposes. Both the production of bismuth and the market price of the metal have been strictly controlled for many years. Messrs. Johnson, Matthey and Co., Ltd., are the chief buyers, and they report official prices per lb. of the metal as follows:—January,

1912, to August, 1914, 7s. 6d.; August, 1914, to March, 1916, 10s.; March, 1916, to January, 1918, 11s.; January, 1918, to January, 1920, 12s. 6d. It is stated that bismuth ore to be marketable should contain at least 10 per cent. metallic bismuth, but it is well known that tinny and other residues containing 5 per cent. have been readily saleable during the last few years.

**Palm Oil as Fuel for Internal Combustion Engines.**—

Major Trentels, of the Belgian colonial army, has recently described some interesting trials of palm oil in a two-cycle semi-Diesel engine of 8-10 h.p. at 500 rev. per min. The only special feature related to the means for keeping the fuel liquid, and this was accomplished by using the cylinder cooling-water in conjunction with the hot exhaust-gases. The engine was started on petrol, and arrangements were made for the termination of the run to be carried out on the same fuel so as to remove palm oil from the feed pipes. The palm-oil consumption was 0.68 to 1.21 lb. per h.p.-hr. for powers between 3.5 to 120 h.p. The calorific value of the fuel was 16,610 B.T.U., and its price was 250 fr. per metric ton (crude petroleum being about 2000 fr. per metric ton). Other vegetable oils have been suggested at various times for use as motor fuel, such as castor oil and groundnut oil, but the advisability of using for fuel any oil which can be utilised for edible purposes is very doubtful.—(*Times Eng. Suppl.*, Feb., 1921.)

**The Chemical Treatment of Seeds.**—Various methods of treating seeds have recently been brought forward in France to improve vegetative growth and to obtain increased yields by steeping them in a solution containing a fertiliser, e.g., potassium nitrate, ammonium sulphate, ammonium phosphate, etc., either singly or in admixture. Investigations by Dr. Worley, of Munich, in 1886 led him to conclude that the steeping of seeds in such a solution has no more effect than immersion in pure water; that germination in such cases is stimulated solely by imbibition; and that solutions of many fertilisers are harmful unless used in very low concentration. Later, M. André, of the Institut National Agronomique, found that most seeds are killed even by a very weak acid solution, and that although many saline solutions facilitate germination when used in concentrations not exceeding 0.2-0.4 per cent., they might be harmful in higher concentrations. Further trials were carried out in France by M. Bachelier, using barley seeds and potassium nitrate, but the results, like those obtained by various Agricultural Services, were inconclusive. The excellent results said to have been obtained by M. Pion-Gaud, who used a 2 per cent. solution of ammonium nitrate, were due less to steeping than to special methods of cultivation. Certain liquids of secret composition, reported to have given favourable results, are now being officially investigated. The general conclusion seems to be that, although germination can be stimulated by steeping the seed in a nutrient liquid, pure water is efficient and preferable.—(*Official.*)

**Guano Deposits on Latham Island, near Zanzibar.**—

The central plateau of Latham Island, a small island situated to the south-east of Zanzibar, is covered with a deposit of guano one foot deep, a sample of which was analysed at the Imperial Institute and found to contain: Citric-soluble  $P_2O_5$ , 19.1% (total  $P_2O_5$ , 29.52%);  $K_2O$ , 0.33%; and  $CaO$ , 35.36%. It resembles the guano obtained from Saldanha Bay, South Africa, and contains more phosphate and less nitrogen than typical Peruvian guano. Freight rates are at present too high to permit of exporting the guano to the United Kingdom, but a market might possibly be found for it in East Africa or Zanzibar.—(*Bull. Imp. Inst.*, 18, 2, 1920.)

**The Effect of the Inhalation of Gases.**—Under this title, Dr. G. A. Welsh summarises his experiences at H.M. Factory, Gretna, in the January issue of the *Journal of Industrial Hygiene*. Volume for volume, says Dr. Welsh, the compounds of sulphur and oxygen are less dangerous than nitrogen oxides or nitrosyl chloride, and sulphuric acid fumes are less harmful than those of nitric acid. From the nature of the substance causing gassing and from observation of the symptoms, it is possible to give a fairly accurate prognosis in all cases except those of gassing by nitrogen peroxide and nitric acid mist. In the majority of severe cases the urgent symptoms subside in 24 hours, although a few last for 48 hours; and it is then a question of dealing with pharyngitis, laryngitis, bronchial irritation or bronchitis. With pharyngitis and bronchitis it is advisable to give the patients a change of air and surroundings for a week before certifying them as fit to return to work.

The wearing of respirators by operatives was discontinued at Gretna after many trials; the operatives dislike them, and as gassing is usually the result of an accident, it is impossible to be sure that the operative will be wearing a respirator at the time of the accident. The only exception is the wearing of moist cotton-wool well over the mouth and nose by nitro-cotton dippers; this cannot be called efficient, and only a self-contained apparatus, such as the "Mecco" has been found to trap nitrogen peroxide fumes effectively. The Mecco provides an atmosphere of oxygen from a cylinder and removes carbon dioxide with caustic soda. Of the 361 cases of gassing which occurred at Gretna, only one—a man who inhaled vapours of fuming nitric acid while working in a closed space—ended fatally.

Systemic poisoning, due to inhaling small quantities of toxic fumes from time to time, can only be cured by removal from work and change of air, when a very simple treatment—light fluid diet and alkaline saline mixture—restores to health. There is no evidence that sound teeth are affected by inhaling harmful gases, but if a tooth is already diseased decay is accelerated. Operatives are warned in every case to pay especial attention to the cleansing of the teeth. The article also describes symptoms and first-aid treatment.

**Chromium Deposits in Serbia and Bosnia.**—The chromium deposit at Raotina, near Skopljé, along the Vardar river, has been worked sporadically in the past. During the war it was exploited by the Germans, who obtained a daily output of about 150 tons. They also worked a second deposit, about 2 kilom. distant from Raotina. Chrome ore was first worked at Dubostica, in Bosnia, in 1884, and during the war the output was about 2000 tons a month. The deposit, which is of considerable extent and width, is owned by the Jugo-Slav Government. Operations are at present suspended, but will be resumed. About 520 t. of high-grade ore was awaiting transportation in June last.—(*U.S. Com. Rep.*, Dec. 20, 29, 1920.)

**Bauxite Deposits in Istria and Dalmatia.**—Little attention was paid to these deposits until during the war, when Germany was obliged to replace supplies formerly derived from France. Exploitation began in 1916, and by the end of 1918, 95,000 metric tons had been exported from Dalmatia and 70,000 t. from Istria. The deposits occur in two zones parallel to the Dinaric Alps, one zone—the most important—being on the mainland and the other on the islands. The ore contains 50–52 per cent.  $\text{Al}_2\text{O}_3$ , 20–24 per cent.  $\text{Fe}_2\text{O}_3$ , and small amounts of silica. Large reserves of ore are said to exist, and as ample water power could be obtained from the river Cherca, the district is suitable for the establishment of an aluminium factory.—(*G. Chim. Ind. ed App.*, Nov., 1920.)

**Iron Ore in Switzerland.**—The iron mines in the vicinity of Gonzen (Toggenburg) produce 80–100 tons of ore daily, which is transported by telfer. As Switzerland has no blast furnaces, the ore is sent to Germany to be smelted. At the present time production has been greatly reduced on account of the crisis in the iron industry.

**The Lignite Mines of Semsaes (Switzerland).**—In 1920 these mines, which are situate near Fribourg, produced 8000 tons of lignite, of calorific power 5500, which were sold at an average price of 22 francs per car-load. Owing to the reduced price of coal, half of the labour employed has been dismissed, and the other half was offered a reduction in wages of 33 per cent., i.e., from 1'20 fr. to 0'80 fr. per hour. The offer was accepted, as the workers knew that if they refused the whole undertaking would be closed down. The men working above ground now work for 10 hours daily.

**Chemical Industry in Czecho-Slovakia.**—Since the foundation of the Czecho-Slovak Republic its chemical industries have appreciably recovered from the depression which prevailed during the war; they are well organised individually and also form part of a larger association, the Federation of Czecho-Slovak Industries at Prague. The chemical factories were mainly erected in Bohemia and Moravia, doubtless owing to the excellent transport facilities afforded by the Elbe and the Moldau. Important fertiliser plants are located at Kolin, Slany, Preran, and Brünn, and those at Slany and Preran also manufacture glue. Pharmaceutical products are made at Chrast, near Chrudim, and in Prague acids and explosives by the Nobel Dynamite Co. at Bratislava, and paints, varnishes, and shellac at Usti (Aussig). Mineral colours, especially ultramarine and Paris blue, chrome, and zinc colours, are made in many Bohemian towns, and coal-tar dyes are produced by the Aussig Chemical Co. at Usti, and by Kinziberger and Co. at Prague. The dye industry is not firmly established, German competition having proved too formidable in spite of a special high tariff. Ink is made by many firms, and at Ceske Budejovice (Budweis) is the famous plant of L. C. Hardtmuth, making pencils and colours. The manufacture of shoe-blackening is rapidly developing chiefly in North Bohemia. Factories producing greases, oils, essential oils, etc., are found chiefly near the large towns; before the war the production of vegetable oils, e.g., linseed oil, was an important industry in Bohemia, but recently it has had to face competition from the cheaper fish and mineral oils. The soap industry has also suffered from foreign competition, and, since the war, from the liquidation of Government supplies of fats and oils; the largest factory is that of G. Schicht, near Usti, which employs more than 4000 workers. Starch and starch-products are made almost entirely in Moravia and the adjoining parts of Bohemia (*cf. J.*, 1920, 17, 275, 306, 400, 438 a).—(*U.S. Com. Rep.*, Jan. 21, 1921.)

**Discovery of Lead-Silver Ore in Italy.**—A rich lode of argentiferous galena has been found in the Alpine part of Valtellina (the upper Adda valley, N. Italy): it is extensive and runs vertically for about 50 ft. into the mountain. About 15 square metres have been opened up, and the ore assays over 65 per cent. of lead, 8 per cent. of silver, with traces of gold and copper.

**The Sugar Situation in Italy.**—The Italian production of beet sugar in 1920 is estimated at 120,000 metric tons, compared with 192,000 t. in the previous year, the reduction being due, it is believed, to the fixing of the price of beets, which made it more profitable to grow other crops. Sugar is rationed at the rate of 1'1 lb. per head per month, but the home production is insufficient, and im-

portation is necessary. During 1919, 73,000 t. was imported, of which the United States supplied 60,000 t., but during the first eight months of 1920 the total importation was reduced to 6500 t.—(*U.S. Com. Rep.*, Feb. 5, 1921.)

**Italian Chemical Periodical Literature.**—With reference to the statement which appeared on p. 22 (1921) of this Journal, we are asked to state that the *Giornale di Chimica Industriale ed Applicata* is published jointly by the Associazione Italiana di Chimica Generale ed Applicata of Rome and the Società di Chimica Industriale of Milan.

## LEGAL INTELLIGENCE.

**SALE OF CHEMICALS IN A "JOB LOT."** *W. Barnes and Co. v. C. Wallington and Co.*

In the King's Bench Division on February 17, Messrs. W. Barnes and Co., London, sued Messrs. C. Wallington and Co., of London, to recover damages for alleged breach of contract.

On behalf of plaintiffs it was stated that, after inspection, they had purchased from the defendants some sulphate of soda and soda ash lying in a warehouse. According to the contract notes there were approximately  $4\frac{1}{2}$  tons of soda ash at £4 per ton and about 40 tons of sulphate of soda in bags at 30s. per ton, but the defendants only delivered 3 tons 12 cwt. of soda ash and 15 tons 3 cwt. of sulphate of soda. Damages were also claimed because some of the material had to be re-bagged. Without hearing evidence for the defence, Mr. Justice Rowlatt gave judgment for the defendants, with costs. It was abundantly clear that the goods sold and bought "as inspected by purchasers" were a heap—a job lot at a low price, and, in the position they occupied, uncountable; the words "about" and "approximately" meant that the amount was only estimated for the purpose of fixing the price. The plaintiffs' claim in respect of re-bagging also failed.

**VALIDITY OF PRE-WAR CONTRACTS.** *E. W. J. Copee and Others v. Blagden, Waugh and Co.*

This action was heard before Mr. Justice Rowlatt in the King's Bench Division on February 17. On behalf of plaintiffs it was stated that they carried on business in Belgium, and that early in 1912 they made nine contracts with the defendants for the sale to them of very large quantities of benzol and naphtha, deliveries to last from the beginning of 1914 to the end of 1918. Plaintiffs now asked for a declaration that the contracts were void on the grounds that (1) under the Proclamation of September, 1915, trading with the plaintiffs was forbidden as they were in territory occupied by the enemy; (2) long delay had occurred; and (3) under the Courts Emergency Powers Act, the Court could annul contracts if it was of opinion that the contracts could not be carried out without serious hardship to the contracting parties. Counsel for the defendant company agreed that the contracts still subsisted by virtue of the clause providing that contracts should be suspended in case of war. He also said that the Act referred to did not apply to Belgians, but only to British subjects.

His Lordship held that there had been complete frustration of the contracts owing to the German occupation of Belgium. The suspensory clause did not provide for such a casualty as that which had overwhelmed the plaintiffs and entirely changed the conditions of the contract. Judgment was given for the plaintiffs for the declaration claimed, with costs.

## PARLIAMENTARY NEWS.

### HOUSE OF COMMONS.

#### Imports from Germany.

In a written answer to Capt. Terrell, Sir P. Lloyd Greame gave the values of the imports from Germany during the third and fourth quarters of 1920 as £8,743,000 and £10,494,000, respectively. The more important increases were as follows:—

	Third Quarter. Tons.	1920.	Fourth Quarter Tons.
Earthenware .. ..	420-6	•	668-4
Glassware, illuminating ..	773-4	•	532-5
Glass bottles .. ..	4380-8	•	5665-4
Lead, pig .. ..	1762	•	3884
Zinc, crude .. ..	1844	•	7671
Potassium nitrate .. ..	12	•	800-4
Silver nitrate .. ..	6-2	•	13-2
Coal-tar dyes .. ..	1186-8	•	2968-4

The values of the coal-tar dyes imported during the two quarters were £1,010,546 and £3,041,798, respectively.—(Feb. 21.)

#### British America Nickel Corporation.

Sir P. Lloyd Greame, in an answer to Mr. Armitage referring to the statements made in the Seventh Report from the Select Committee on National Expenditure, said that the British America Nickel Corporation, in which the Government had invested \$3,000,000 (*cf.* J., 1918, 142), had put forward a scheme to raise further working capital and to modify the rights attaching to the various capital issues of the Corporation. No further liability was imposed on the Government by the scheme, which offered the best prospects of a satisfactory realisation of the investment.—(Feb. 21.)

#### The Dyestuffs (Import Regulation) Act.

In reply to Major Barnes and Mr. Waterson, Sir P. Lloyd Greame said that no decision had been made to restrict the importation of goods under the Dyestuffs Act to those for which the applicant had a definite order, but it would facilitate the granting of licences if it were clear that the dyestuffs were ordered for persons who required them. A complete list of commodities covered by the Act would be available shortly, and information as to the quantities of dyestuffs and other products licensed under the Act would be published periodically; no useful purpose would be served by publishing a list of the firms that had received licences. The refusal to grant an import licence for 2 cwt. of salicylic acid (B.P.) for pharmaceutical purposes would be reconsidered if the applicants could satisfy the Dyestuffs Advisory Licensing Committee that the consignment was of B.P. quality, and intended solely for medicinal use, and if they would undertake to prove later that it had been so used.—(Feb. 21.)

#### Food Values.

Dr. Addison informed Capt. Elliott that the publication of a pamphlet giving information on the dietetic values of foods was being considered, and certain researches relating thereto were being conducted in collaboration with the Medical Research Council.—(Feb. 25.)

#### Imports of Gas Mantles.

In a written reply to Sir J. Norton-Griffiths, Sir R. Horne stated that 46,938 gross (value £101,973) of incandescent gas mantles was imported during the six months ended January 31, 1921, and that this quantity was small compared with that in pre-war years. He was aware of the difficulties experienced by the British industry through lack of orders.—(Feb. 28.)

*Foreign Competition in the Coal Trade.*

Answering Lieut.-Col. Sir F. Hall, Mr. Bridgeman said that the United States exported 476,000 tons of coal to Europe in the year ended June, 1913, and 13,383,000 t. in the calendar year 1920; the coal exported from Australia to Europe was 100 t. in 1913 and 113,000 t. in 1920. The British output of coal during the last six weeks of 1920 seemed to show that a further 13 million tons might have been produced during the year without employing additional labour.—(Feb. 28.)

*Sugar Industry (Foreign Competition.)*

Mr. Chamberlain, in a written reply to Capt. Bowyer, said that all restrictions on the import of sugar were removed on February 26, after full discussion with the refiners. Apprehensions regarding the effect on the sugar industry of competition from countries enjoying cheaper labour and a favourable exchange were premature, but no statement as to future legislation could be made.—(March 1.)

*The Key Industries Bill.*

Sir P. Lloyd Greame, replying for the Prime Minister to Mr. Terrell, announced that the above Bill would be introduced and proceeded with immediately after Easter.—(March 9.)

## REPORTS.

GENERAL REPORT ON THE INDUSTRIAL AND ECONOMIC SITUATION OF NORWAY IN DECEMBER, 1920. By C. L. PAUS, *Commercial Secretary to H.M. Legation, Christiania. Department of Overseas Trade.* Pp. 75. London: H.M. Stationery Office. 1921. [Cmd. 1145. 9d.]

The previous report on Norway (*cf. J.*, 1920, 364B), which covered the war-period down to the end of 1919, has now been supplemented by a general report for the year 1920.

Norway, like other nations which knew prosperity during the war, has now become engulfed in the world-wide depression. The brighter prospects which were apparent in the early months of 1920 disappeared quickly, and the slump which followed has gradually extended to nearly every branch of industry and commerce; labour problems, social unrest, financial stringency, and an abnormal exchange, all contribute to the general depression. The balance of trade is very adverse, and much capital is locked up in stocks. German exporters, favoured by the conditions of exchange, can now undersell all competitors, and are in fact offering their goods, including drugs and chemicals, at very low prices for prompt delivery. Several large German firms have opened offices in Christiania.

The market for heavy chemicals was (in December) very uncertain, and owing to the curtailment of production by the pulp and paper mills, demand was practically non-existent. America sent considerable supplies during the year, and at the close Germany was offering cheap goods to an already glutted market. Many of the factories erected at great expense during the war cannot pay dividends on the invested capital, and with few exceptions production has been greatly curtailed. The carbide industry has suffered from the shortage and high price of imported anthracite; two factories have closed down and the rest are working at 30–40 per cent. of their capacity. The ferro-silicon works are in a still worse position, and the demand for aluminium and refined zinc, which was good in the early part of the year, has diminished.

Mining has been hampered by dear labour and fuel, high freights, and difficulty in obtaining payment from Germany. Production of iron ore has ceased, and pyrites mining is very restricted; it does not pay now to extract the copper from cupreous pyrites owing to the low price of the metal. Nickel, chromium, and molybdenite mines remained closed throughout the year. In the paper-making industry, Finnish and German competition is being felt; the boom which prevailed at the beginning of the year has passed, demand is negligible, and the mills have cut down their production to one-third or less. Exceptionally, the margarine industry has escaped the prevailing depression; demand is described as normal, but profits, owing to competition, will be small. Soap-makers, too, have been busy, and as they are independent of imported oils, they can undersell foreign manufacturers. The nitrate factories controlled by the Norwegian Hydro-electric Nitrogen Co. and the Cellulose and Mechanical Pulp Mills are reported to have been fully occupied throughout the year and to have sold their output some months ahead; it is, however, anticipated that prices will fall.

REPORT OF THE DEPARTMENTAL COMMITTEE ON CONTROL OF CERTAIN THERAPEUTIC SUBSTANCES. *Ministry of Health.* Pp. 13. H.M. Stationery Office. 1920. [Cmd. 1156. 2d.]

This Committee was appointed by the Minister of Health in April, 1920, "to consider and advise upon the legislative and administrative measures to be taken for the effective control of the quality and authenticity of such therapeutic substances offered for sale to the public as cannot be tested adequately by direct chemical means." The substances referred to are classified as follows:—A. Such products as vaccines, sera, toxins and anti-toxins; B. Synthetic chemicals, such as salvarsan and its analogues; and C. Galenic and other preparations administered through the mouth, such as digitalis, strophanthus, squill, ergot, etc. Substances in A and B are not included in the British Pharmacopœia; the standardisation of those in A is left at present entirely to the manufacturer, but those in B are subject to a measure of Government control, *e.g.*, the Board of Trade issues licences for the manufacture of salvarsan, attached to which are conditions relating to maximum prices and testing by the Medical Research Council. Most of the substances in C are included in the B.P., but it is held by many that the standards therein prescribed are inadequate.

The recommendations of the Committee include the following:—Therapeutic substances which cannot be tested adequately by chemical means should be subject to supervision and control; the controlling authority to be the Committee of the Privy Council appointed under the Ministry of Health Act, 1919, assisted by an advisory committee. A central laboratory should be established under the Medical Research Council for preparing standards, conducting research, and testing samples taken from manufacturers' stocks or bought in the open market. Manufacturers should be licensed, and their plant, premises and processes inspected, but in the case of substances in B and C, inspection should be confined to the records and methods of biological testing, and, when necessary, to the filling and sealing of the containers. Similar restrictions should apply to products made abroad. Imported goods must fulfil the same conditions as to purity, etc., as home-manufactured goods; they should bear an approved form of label, and come under Section 42 of the Customs Consolidated Act of 1876. Fresh legislation would be necessary to give effect to these recommendations.

## OFFICIAL TRADE INTELLIGENCE.

(From the Board of Trade Journal for February 24 and March 3.)

### OPENINGS FOR BRITISH TRADE.

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W. 1, from firms, agents, or individuals who desire to represent U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number:—

Locality of Firm or Agent.	Materials.	Reference number
South Africa ..	Glassware, paper ..	273
" ..	Druggists' sundries, disinfectants, fertilisers ..	274
" ..	Paint, varnish, crockery ..	250
Denmark ..	Non-ferrous metals, iron tubes ..	277
France ..	Industrial chemicals, drugs ..	279
" ..	Drugs ..	280
Italy ..	Heavy chemicals, copper sulphate, caustic soda, soda crystals, fertilisers, oils, fats ..	281
" ..	Chemicals, non-ferrous metals ..	282
Rumania ..	Sugar, olive oil, soap ..	283
Spain ..	Non-ferrous metals ..	284
Sweden ..	Paint ..	286
United States ..	Raw materials for paint manufacture, dyes for textiles ..	289
Mexico ..	Black and galvanised pipes, corrugated roofing, wall and floor tiles, sanitary ware ..	266
Argentina ..	Lubricating oils and greases ..	292
" ..	Earthenware ..	293

### TARIFF, CUSTOMS, EXCISE.

**Belgium.**—Beet syrup may now be exported within limits to be fixed by the Ministry of Industry, Labour and Supplies.

**British Guiana.**—A copy of the new Preferential Customs Tariff may be seen at the Department. Among the articles that are admitted at half rates under the British Preferential Tariff are certain kinds of iron and steel, common soap, fertilisers, vermin killers, insecticides, and patent medicines containing not more than 50% of proof spirit.

**British Honduras.**—It is proposed to reduce the export duty on all alcoholic beverages to 5 per cent. *ad valorem*.

**British India.**—The tariff valuations of exported raw hides and skins have been revised with effect from February 5.

**Chile.**—It is proposed to exempt from increased customs duties condensed and dried milk, cocoa, sugar, petrol, and oils.

**Denmark.**—Import prohibitions have been removed from nitric, oxalic, and sulphuric acids, nitrates, chlorates, oxalates, perchlorates, glycerin, carbolic acid, aniline benzene, toluene, naphthalene, cymol, sulphur, phosphorus, iodine, mercury, sodium, potassium, aluminium powder, calcium phosphide, antimony sulphide, lead dioxide, and manganese dioxide.

Export prohibitions have been withdrawn from petroleum, benzene, fuel oil, and chocolate.

**Egypt.**—The prohibition of the import of sugar is extended to February 1, 1922.

**Finland.**—All exports except grain and sugar are now entirely free from licence.

Among the articles the import of which is prohibited except under licence are meat extracts, cocoa powder, chocolate, sugar, molasses, glucose,

honey, saffron, vanilla, vanillin, mineral waters, silk, certain papers and paper manufactures, certain leather wares, many metal wares (except platinum wares for scientific or technical use), certain wares of porcelain, earthenware and glass, toilet soap, certain essences, and essential oils.

**France.**—Until further notice the export is permitted, without special authorisation of organic and chemical fertilisers, ammonium sulphate, nitrates of lime and soda, dephosphorisation slag, superphosphate of lime, calcium cyanamide, and undressed hides and skins.

Recent customs decisions affect the classification of, *inter alia*, anodes of molybdenum, artificial slates or tiles of asbestos and cement, cardboard cut for packing margarine, pneumatic tyres for side-cars, anti-friction fibres of lead impregnated with plumbago, alloys of magnesium and other common metals, artificial dolomite, lanolin, nickel, and residues of vegetable oils produced in the manufacture of edible fats.

**France and Algeria.**—The prohibition of the export of and the export duty on spirit of turpentine have been withdrawn as from February 12.

**French West Africa.**—"Coefficients of Increase" have been established on certain imports and exports, including kola nuts, sugar, alcohol, salt, silver, matches, groundnuts, palm kernels, palm oil, and gum arabic.

**Germany.**—The import of raw cocoa, tallow, certain oil fruits, and oil-seeds is permitted without licence.

The import and export of sulphur are permitted without licence as from February 23.

**Gold Coast.**—The new schedule of import duties came into force on January 22. Among the articles affected are alcoholic beverages, candles, cement, lard, lard compound, lead, matches, mineral oil, soap, and sugar. Drugs and medicinal preparations recognised by the B.P. and mineral fuel oil are admitted free of duty.

**Hungary.**—Among the articles on which the customs duties are temporarily suspended are sugar, molasses, colza seed, rape seed, butter, lard, goose fat, margarine, fish oil, seal oil, animal and vegetable tallow, palm oil, palm kernel oil, coconut oil, pyrites, mineral oils, lignite and schist tars, and crude lead and zinc.

**Italy.**—The import of crude and refined mineral oils is free from restriction, but the "aales" tax is to be maintained.

The surcharge on customs duties when paid in paper currency is fixed at 300 per cent. as from February 15.

**Latvia.**—Recent customs decisions affect linseed, coconut oil, edible vegetable oils, margarine, cocoa butter, and carborundum wheels.

**New Zealand.**—Gelatinised pigmented paper to be sensitised before use with a solution of potassium dichromate may be imported duty free under the British Preferential Tariff.

**Poland.**—Among the articles on which the import duty is suspended until April 1 are condensed and dried milk, margarine, fertilisers, train oil, graphite, ammonium nitrate and sulphate, Glauber's salt, sulphuric, nitric, and hydrochloric acids, dye-earths, pig iron, antimony, copper ingots, lead blocks and ingots, certain kinds of paper, and cotton wool.

The customs duty has been reduced on, *inter alia*, sugar, olein, textile leather, asbestos, ammoniacal soda, soda crystals, sodium sulphide and hydrosulphite, nitro- and amino-aromatic compounds, naphtho- and sulpho-derivatives, ether, certain metals and metal manufactures, and wood pulp. On all other goods the "agio" has been raised to 1900 per cent.

**Rumania.**—Export duties have been removed from carbide, carbon disulphide, calcium hypochlorite and chloride, and gypsum.

**Serb-Croat-Slovene State.**—The import of sugar is free from all restrictions but is subject to a "monopoly" tax of 200 dinars per 100 kg., in addition to other taxes.

**Tunis.**—A general sanction has been given for the export of 2250 metric tons of olive oil to foreign countries.

## COMPANY NEWS.

**ANGLO-PERSIAN OIL CO., LTD.**—A special resolution was passed at an extraordinary meeting held on February 22 authorising, *inter alia*, (1) the conversion of the existing 6 per cent. cumulative participating preference shares into 8 per cent. cumulative non-participating first preference shares; (2) the repayment of capital to the holders thereof, in the event of liquidation, in priority to other issues, plus a minimum of 10 per cent.; (3) further issue of shares up to a maximum of £10,000,000. Sir Charles Greenway explained that the new capital was required to carry out the company's extensive programme of development. Net profits, which for the year ended March 31, 1920, amounted to £2,811,615, would not be less than £4,000,000 for the year ending on March 31 next. The company was particularly fortunate in possessing very productive wells worked at an extraordinarily low cost, and oil which contained an unusually high proportion of benzine and kerosene.

**BRADFORD DYERS' ASSOCIATION, LTD.**—The address of Sir Milton S. Sharp, delivered at the ordinary general meeting held on February 28, was devoted to a discussion of the general industrial situation and suggestions for improving it. The dyeing industry is most seriously affected by the great cost of coal, and the difficulties of the textile trade in this country are very much greater than in America because some 75 per cent. of our production is for export and the present state of the exchanges almost excludes the possibility of foreign trade; in the United States the ratio of exports to production is relatively very small. The profits of the Association for 1920 were slightly higher than for 1919, but owing to the necessity of meeting the depreciation in the company's investments, mainly Government securities, the total dividend for the year, 20 per cent., is 2½ per cent. less than that paid last year.

**LEVER BROS., LTD.**—At an extraordinary meeting held at Liverpool on February 28 it was resolved to create £15,000,000 of redeemable first mortgage debenture stock, and to make an immediate public issue of £4,000,000 of such stock. After referring to the impossibility of raising money at the present time by issuing shares, Lord Leverhulme said that during last year the company had purchased ordinary shares in the Niger Co., J. J. Crosfield and Sons, W. Gossage and Sons, Prices' Patent Candle Co., and John Knight, and, with the exception of the shares in the Niger Co., the company had realised at a profit all the stock taken over from the others. The financial assistance given to many of the associated companies made heavy calls on the company's resources, and money was also needed for the ordinary conduct of the business. The soap trade was suffering less from the prevailing depression than many other trades, but all new development work had been stopped, and until the debentures were cleared or greatly reduced the directors would not enter upon any new undertaking or acquire further capital.

**COURTAULDS, LTD.**—The directors' report states that the year 1920 was one of extraordinary vicissitudes; during the greater part of it business exceeded all records and the company's productive capacity was strained to the utmost, but towards the end this situation was reversed and production had to be restricted. In particular the textile department was seriously affected by the importation of foreign silk goods. During the year the share capital of the company was raised from 4 to 12 millions sterling by the capitalisation of the reserve account. Profits, after deducting all expenses and taxes, amounted to £1,804,796, which compares with £2,280,861 in 1919 and £1,184,938 in 1918. Dividends for 1920 total 12½ per cent., free of tax, on the new capital, which is equivalent to 36½ per cent. on the capital as in 1919, when the distribution was 30 per cent.; £200,000 is placed to reserves, against £999,993, and £383,874 is carried forward, compared with £229,077 brought in. The present value of the company's holding in the Viscose Co. is stated to exceed the valuation placed upon it in the balance-sheet. The course of business in America in 1920 was similar to that in this country, but improvement has recently set in.

## GOVERNMENT ORDERS AND NOTICES.

**PROHIBITED EXPORTS.**—The Board of Trade (Licensing Section) announces that, as from March 1, 1921, lard has been removed from Lists A and B of Prohibited Exports. Also that the following new headings have been substituted for existing headings:—

(A) Opium and any preparation thereof containing not less than 0·2 per cent. of morphine or 0·1 per cent. of diamorphine; (A) cocaine and salts thereof, and any substance containing not less than 0·1 per cent. of cocaine; (A) diamorphine and salts thereof, and any substance containing not less than 0·1 per cent. of diamorphine; (A) ecgonine and salts thereof, and any substance containing not less than 0·1 per cent. of ecgonine; (A) morphine and salts thereof, and any substance containing not less than 0·2 per cent. of morphine.

**PROJECTED NEW RESEARCH ASSOCIATIONS.**—The Department of Scientific and Industrial Research notifies that the formation of associations for the jute and cast-iron industries has been approved, and that the memoranda and articles of association for two others dealing respectively with aircraft and liquid fuels for oil-engines are under consideration.

**COMMITTEE ON PATENTS.**—The Lord President of the Council has appointed an Inter-Departmental Committee on Patents (1) to consider the methods of dealing with inventions made by workers aided or maintained from public funds, whether such workers be engaged (a) as research workers, or (b) in some other technical capacity, so as to give a fair reward to the inventor and thus encourage further effort to secure the utilisation in industry of suitable inventions and to protect the national interest; and (2) to outline a course of procedure in respect of inventions arising out of State-aided or supported work, which shall further these aims and be suitable for adoption by all Government Departments concerned.

The sixteen members of the Committee include Mr. Kenneth Lee (chairman), Dr. H. H. Dale (Head of the Department of Biochemistry and Pharmacology, Medical Research Council), Sir Frank Heath, and the Hon. Sir Charles A. Parsons. The secretary is Mr. A. Abbott (16 and 18, Old Queen St., S.W.1).



## TRADE NOTES.

## BRITISH.

**Ceylon in 1919.**—In spite of difficulties due to freight and rise in exchange, there was a general revival of trade in 1919. The imports, of which the U.K. supplied about 14 per cent. and British India and Burma over 54 per cent., rose in value from £11,847,784 in 1918 to £15,954,915 in 1919 (the value of the rupee being taken at 1s. 4d.). The most notable increases were in fertilisers, metals and metal-ware, and cement; of the last-named the U.K. supplied 173,119 cwt. and Japan, a keen competitor, 126,913 cwt. Exports, valued at £24,470,319, rose considerably, the chief increases being in tea, rubber, desiccated coconuts (five times the value of the export in 1918), copra, and coconut oil; the United Kingdom took over 42 per cent. and the U.S.A. over 33 per cent. (nearly double the percentage in 1918). The increase in the export of rubber, from 21,079 tons in 1918 to 44,818 t. in 1919, was partly due to stocks accumulated in 1918 owing to freight restrictions. The export of cacao decreased from 79,025 cwt. in 1918 to 54,742 cwt. in 1919, but that of cardamoms rose by nearly 50 per cent. to 561,244 lb.

In the mineral industry, there was a very sharp decline in the production of salt, the output of 319,463 cwt. comparing with 1,186,524 cwt. in the record year 1918, and representing only about half the annual consumption. Graphite mining has steadily declined since 1917, and is now at a very low ebb; 50 mines were working in 1919, compared with nearly 1300 in 1917, and the exports have decreased from over 33,000 t. in 1916 to 6671 t. in 1919. The position is viewed with anxiety, as the present slump, unlike previous slumps, is due not to lack of demand for graphite, but to supplies being obtained elsewhere. The few mines now working are producing more than the requirements, thereby adding to the existing large stocks. The attempt to revive mica mining failed, as the deposits are too small for profitable working, although some amber mica fetched good prices. The production of monazite sand on a small scale was continued at Indruwa. Materials suitable for the manufacture of Portland cement were discovered near Jaffna, and it was hoped that a cement works might be established. Sub-committees of the Industries Commission inquired into the production of hydro-electric power, for which favourable conditions are said to exist, and into glass, soap, sugar, tanning, vegetable-oil and other industries.—(*Col. Rep. Ann., No. 1049, Nov., 1920; U.S. Com. Rep., Jan. 12, 1921.*)

**Egyptian Imports of Heavy Chemicals.**—Practically all the heavy chemicals imported into Egypt are supplied by the United Kingdom, with the exception of zinc oxide, which is obtained mainly from Belgium. The imports of heavy chemicals during the first eight months of 1920 comprised:—Potassium and sodium bichromate, 202 long tons; nitrate of soda, 55,500 t.; sal ammoniac 198 t.; zinc oxide, 396 t.; caustic soda, 1500 t.; soda crystal and soda ash, 70 t.; glue, 88 t. The caustic soda imported included 33 t. from the United States, which was the only item of any importance supplied by that country.—(*U.S. Com. Rep., Feb. 1, 1921.*)

## FOREIGN

**The Celluloid Trade of Siam.**—Celluloid goods are not manufactured in Siam, and the imports have been comparatively small during the past four years. The trade is practically in the hands of Japanese manufacturers who supplied 90 per cent. of the imports in 1919–1920, compared with 1.6 and 1.4 per cent. from the United Kingdom and the United States, respectively.—(*U.S. Com. Rep., Dec. 31, 1921.*)

**Dutch Trade in Oils and Fats in 1920.**—During the past year Dutch interests have acquired large holdings in German and Austrian margarine undertakings, so that in the future Holland is likely to become a clearing house for Central Europe in these trades. Although the high exchange value of the American dollar told against American exports and favoured those from Argentina, the United States nevertheless supplied four-fifths of the Dutch imports of lard, nearly all the neutral lard, about three-fourths of all other animal fats, 93 per cent. of the raw margarine, and 45 per cent. of the cottonseed oil. Difficulty was experienced in obtaining extra-quality oleo oil from the United States. Germany took 37 per cent., and Great Britain 50 per cent. of the margarine exported, and 55 and 25 per cent., respectively, of the coconut oil.—(*U.S. Com. Rep., Jan. 26, 1921.*)

**Credit Organisation in the German Nitrogen Industry.**

—In order to encourage the purchase of nitrogenous fertilisers, the fertiliser manufacturing groups are now forming large credit organisations. It is understood that a kind of Guarantee Institute in the form of a Stickstoff-Kredit G.m.b.H. will be founded, with an original capital of 500 million marks, in which shares will be taken by the Nitrogen Syndicate and by the various associations of manufacturers, e.g., the Deutsche Ammoniak-Verkaufs-Vereinigung, Bochum, the Economic Federation of German Gasworks, the Upper Silesian Coke Works, and the two firms which manufacture synthetic nitrogen products, viz., the Badische Anilin- und Soda-fabrik and the Bayerische Stickstoff-werke A.-G.

It is intended to induce farmers to purchase and accept delivery of nitrogen fertilisers in the early spring instead of in the late autumn, the Nitrogen Syndicate demanding only one-half of the purchase price on delivery, the rest to be paid by bills of exchange, which will first become due when the farmer receives the profit from his harvest. By this means no interest on the bills of exchange will be required from the nitrogen consumer for several months, and for the rest of the period he will be charged only a low rate of interest.—(*Ed. of Trade J., Mar. 3, 1921.*)

**Nicaragua in 1918 and 1919.**—Nicaragua, the largest of the Central American Republics, has an area of 49,200 sq. miles and a population of over 500,000. It has immense but largely undeveloped resources, including minerals (of which gold only has been worked), mahogany, dyewoods, cabinet woods, and vast stretches of uncultivated agricultural land. The production of gold, sugar, cacao, and tobacco constitute the chief industries; small amounts of soap, leather, etc., are manufactured, but only for local needs. The imports in 1918 amounted in value to £1,185,960, and included:—Cement, £4738; chemicals, drugs and medicines, £52,115; paper and manufactures of, £26,417; petroleum, £30,459. The exports, worth £1,550,988, included: Cacao, £9934; coconuts, £5460; dyewood and dyes, £2048; gold, £249,811; hides and skins, £63,266; rubber, £8523; sugar, £148,415; and eilver, £45,328.

In 1919 the imports were valued at £1,582,530, of which 84 per cent. came from the United States and 9 per cent. from Great Britain; they included:—Chemicals, drugs and medicines, £89,637; cement, £6923; vegetable fibres, and manufactures of, £66,576; paper and manufactures of £31,020; petroleum, £26,087. The exports, worth £2,481,894, comprised:—Cacao, £6296; coconuts, £10,372; gold, £299,190; hides and skins, £83,748; lard, £5524; rubber, £17,181; sugar, 3847 metric tons, £123,759; and silver, £53,504; 62 per cent. of the exports went to the United States, 28 to France, and 4 to Great Britain.—(*U.S. Com. Rep., Suppl., July 13, Dec. 31, 1920.*)



## REVIEWS.

THEORETICAL AND PRACTICAL  
INSTRUCTION IN ORGANIC CHEMISTRY.

A TEXT-BOOK OF ORGANIC CHEMISTRY. By A. F. HOLLEMAN. Fifth English edition, edited and completely revised by H. JAMIESON WALKER and O. E. MOTT. Pp. xviii+642. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1920.) Price 18s. 6d. net.

LABORATORY MANUAL OF ORGANIC CHEMISTRY. By HARRY L. FISHER. Pp. 331. (J. Wiley and Sons, and Chapman and Hall. 1920.) Price 12s. 6d. net.

The continuous growth of organic chemistry induces in many minds a feeling of repletion which occasionally finds utterance in a protest against the addition of further organic compounds to an already enormous list. It is sometimes pointed out that many of these substances appear to have no significance in a general purview of the subject and are often only of passing interest even to the workers who synthesise them with so much persistent industry. Yet, on the other hand, there are several classical instances of organic compounds buried in neglected corners of "Beilstein" which in the long run have proved to be substances of great practical or theoretical importance.

The aromatic arsenical derivative, atoxyl, is one example of these chemical "Cinderellas," and tri-*ac*-dipyridyl ferrous bromide, utilised by Werner in demonstrating the asymmetric iron atom, is another.

It cannot, however, be denied that the existence of this prodigious total of organic compounds imposes a great responsibility on those who compile a students' manual of organic chemistry, where the object in view is a concise and comprehensible presentation of the general principles of the science illustrated by a judicious selection of characteristic types of organic derivatives. The skill of the compiler is to be gauged by his choice of representative examples and by his inclusion within a reasonable compass of adequate references to all the chief divisions of the subject.

That Professor Holleman's well-known text-book has fully met this acid test is sufficiently attested by the fact that the work under review is the fifth English addition, and that 33 editions have appeared in other languages. One characteristic of this revised edition is the inclusion of a large number of data dealing with physical methods of determining the molecular complexity and constitution of organic compounds or of studying the conditions under which these substances interact with one another and with inorganic reagents.

The aliphatic compounds receive adequate treatment, but possibly some compression might be made in this section in order to allow a little more space for the discussion of aromatic derivatives. The three diaminobenzenes are aromatic bases of outstanding importance, but there is only one brief reference to ortho-phenylenediamine (p. 479) and no mention of the interaction of the diamine and nitrous acid. Yet since the production of an ortho-diazoimine is the simplest way of determining the constitution of an aromatic compound containing two contiguous amino-groups, this reaction is of general interest.

In an account of the formation of the two hydroxides from crystal violet by addition of alkali (p. 545) the explanation is obscured by faulty terminology. The first product, a quaternary ammonium hydroxide, is the true colour base, and this designation should not be given to an isomeric colourless substance which is not merely a base but a carbinol. On the whole, however, the nomenclature, which has been carefully edited by the translators,

conforms to the system adopted by the Chemical Society, a concordance likely to be of the greatest help to English students. The increasing difficulty of separating completely the descriptions of open-chain and cyclic compounds is exemplified by the inclusion of geraniol among the unsaturated aldehydes and ketones (p. 177), whereas it seems preferable to place this aldehyde among the terpenes (p. 525). The formula for  $\beta$ -naphthoquinone, which was correct in the last edition, has now become distorted (p. 558), but otherwise the treatise is remarkably free from typographical errors. The work, which has been thoroughly brought up to date, is to be recommended strongly as an unerring guide to modern organic chemistry.

Although "organic preps." are for many students the most attractive section of practical chemistry, it is doubtful whether they inculcate sufficiently those habits of tidiness, accuracy, and practical dexterity which are the best foundation of a training in the laboratory arts. When carried out in a casual and thoughtless manner, organic preparations become merely a hedonistic pastime without the mental and moral training gained in a course of chemical analysis. This danger has evidently been in the mind of Dr. H. L. Fisher in devising his course of laboratory experiments in organic chemistry, for he lays great stress on the accurate determination of carbon, hydrogen, and nitrogen in organic compounds. The author traces the gradual evolution of these methods of organic analysis, to which he has added important developments. He favours the use of an electrically heated furnace both for carbon and hydrogen estimations and for the determination of nitrogen. His employment of alumina as the absorbent for water in place of calcium chloride or concentrated sulphuric acid is an improvement likely to come into general use. These descriptions of the analytical processes occupy about one-third of the book, and attention paid to these very practical details will certainly conduce to success in organic estimations. The remainder of the treatise is devoted to laboratory experiments, including the preparation of a typical series of organic compounds, exercises on distillation, and precise determinations of melting and boiling points. The "quizzes" which form such a characteristic feature of American teaching are very much in evidence at the end of each experiment. The student who tries conscientiously to answer all these searching questions will, by the end of the course, have gathered a very considerable fund of information regarding the nature and behaviour of organic substances. The book includes many references to original literature and to other text-books. Certain of the exercises suggested are somewhat off the beaten track, such as the hydrolysis of hutter, the preparation of lecithin and *l*-menthone, and several typical syntheses in the camphor series. These innovations are a welcome change from the more stereotyped preparations and lend additional interest to an original and inspiring manual of practical chemistry.

G. T. MORGAN.

THE TESTING OF DYE-STUFFS IN THE LABORATORY. By C. M. WHITTAKER. Pp. 100. (London: Heywood and Co., Ltd. 1921.) Price 12s. 6d. net.

Although there were already numerous text-books on dyeing in existence, there has not previously been a treatise dealing with the subject so thoroughly from the point of view of the laboratory dyer. This book will undoubtedly be of great value to those engaged in experimental dyeing on a laboratory scale, and also to those who have to interpret the results obtained in the laboratory.

The first chapter deals in an excellent manner with the equipment and functions of a dyeing

laboratory, and contains a large amount of information based on the author's twenty years' experience. The next seven chapters deal with the dyeing of various classes of dyestuffs in the laboratory, and also with the tests to which the dyeings should be subjected and the results which may reasonably be expected. The treatment is fairly full and eminently practical. A useful chapter on the dyeing of mixed fabrics and a final chapter containing miscellaneous information follow. A series of tables and a full index complete the book.

It is unfortunate that more care has not been given to a revision of the English used in this book, particularly in view of the critical review of the author's previous book (*cf. J.*, 1919, 38, 35 n). A sentence such as "Enter the material cold, raise carefully to boil in half-hour, and dye at the boil half-hour" (p. 11), may be satisfactory in a laboratory note-book, but it should not be allowed to appear in a work of this nature. Again, it is interesting to note that "The water in the dyebath should have had the oxygen taken out of it by adding hydrosulphite powder conc. . . ." (p. 63). Another notable point is the failure of the author to use hyphenated words, which leads to the frequent occurrence in the text of such words as "dyetest." In other parts of the book, the language used is certainly forcible, but by no means elegant. A point of a different character is that the author fails to emphasise sufficiently the necessity of using a freshly-prepared dye-bath as a standard in dyeing most of the anthraquinone vat-colours.

The book is essentially practical, and should be in the hands of all who wish to be acquainted with the peculiar technique of the laboratory testing of dyestuffs.

F. W. ATACK.

A SYSTEM OF PHYSICAL CHEMISTRY. By W. C. McC. LEWIS. VOL. II., THERMODYNAMICS. Third edition. Textbooks of Physical Chemistry. Pp. viii.+454. (London: Longmans, Green and Co. 1920.) Price 15s. net.

The importance of thermodynamics in many branches of industrial chemistry is now admitted; the work of Haber on the synthesis of ammonia is but one of the recent highly successful researches undertaken with an understanding of this branch of physical chemistry. The records of technical chemistry contain more than one example of costly blunders which could have been avoided by an elementary knowledge of thermodynamics.

The applications of thermodynamics to chemistry are so peculiar and specialised that a distinct treatment is necessary; a book written from the engineering standpoint is of little service to the chemist. The difficulties of acquiring a knowledge of the science have been greatly exaggerated and perpetuated by tradition among chemists. Two methods of treatment suggest themselves. Either particular attention is paid to general principles, their meaning and fields of application being considered broadly, or else the fundamental laws and equations are regarded as something to be got over as quickly and painlessly as possible, so that after a little mental leapfrog the far-reaching fields of special applications are entered and the consideration of details begun.

Prof. Lewis has perhaps tended to go too lightly over the fundamental parts of the subject. He has treated a large mass of detailed applications in some cases less critically than he might with advantage have done. In the lengthy discussion of Tinker's contribution to the theory of osmotic pressure, for example, he might well have pointed out that the application to intermolecular volumes of equations deduced for matter in bulk is outside the region of legitimate application of these equations.

In the summary of recent American work on solutions (which is presented without any attempt to connect it with the rest of the book), the reader might have been informed that it is impossible by a really legitimate use of thermodynamics to arrive from the same set of data at two diametrically opposite conclusions.

The sections on electrochemistry, affinity, and Nernst's heat theorem are the most satisfactory parts of the book, and these should appeal especially to technical chemists. A fair knowledge of the calculus is naturally assumed, and all chemists who are so equipped will find Prof. Lewis's book both interesting and useful. Thermodynamics is not of purely academic interest, as the Haber factories in Germany eloquently testify.

J. R. PARTINGTON.

## PUBLICATIONS RECEIVED.

ANTHRACENE AND ANTHRAQUINONE. By E. DE BARRY BARNETT. Pp. 436. (London: Baillière, Tindall and Cox. 1921.) Price 27s. 6d.

SILICA AND SILICATES. By J. A. AUDLEY. *Industrial Chemistry Series*, edited by S. RIDEAL. Pp. 374. (London: Baillière, Tindall and Cox. 1921.) Price 15s.

TABLES OF PHYSICAL AND CHEMICAL CONSTANTS AND SOME MATHEMATICAL FUNCTIONS. By G. W. C. KAYE and T. H. LASY. Fourth edition. Pp. 161. (London: Longmans, Green and Co. 1921.) Price 14s.

ESTATE RUBBER: ITS PREPARATION, PROPERTIES, AND TESTING. By DR. O. DE VRIES. Pp. 649. (Batavia: Ruygrok and Co. 1920.) Price 20fl.

COCOA AND CHOCOLATE, THEIR CHEMISTRY AND MANUFACTURE. By R. WHYMPER. Second edition, revised and enlarged. Pp. 568. (London: J. and A. Churchill. 1921.) Price 42s.

AMMONIA AND THE NITRIDES. By E. B. MAXTED. Pp. 116. (London: J. and A. Churchill. 1921.) Price 7s. 6d.

PROBLEME UND AUFGABEN DER NAHRUNGSMITTEL-CHEMIE. By DR. O. EICHWALD. Pp. 99. (Dresden and Leipzig: Th. Steinkopff. 1921.) Price (unbound) 4s. 5d.

LES VERNIS. By CH. COFFIGNIER. Preface by M. HALLER. *Encyclopédie de Chimie Industrielle*. Pp. 640. (Paris: J.-B. Baillière et Fils. 1921.) Price 40 francs.

ANALYSIS OF PAINT VEHICLES, JAPANS, AND VARNISHES. By C. D. HOLLEY. Pp. 203. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1920.) Price 13s. 6d.

THE ASSAY OF COAL FOR CARBONISATION PURPOSES: A NEW LABORATORY METHOD. By THOMAS GRAY and J. G. KING. *Fuel Research Board Technical Paper No. 1*. Pp. 13. Department of Scientific and Industrial Research. (London: H.M. Stationery Office. 1921.) Price 6d.

TRINITROBENZENE AND TRINITROTOLUENE. By CAPT. M. KOSTEVITCH. Introduction by W. MACNAB. Pp. 38. (London: Millard and Co. 1920.)

UNITED STATES GEOLOGICAL SURVEY. DEPARTMENT OF THE INTERIOR. (Washington: Government Printing Office. 1921):—

FOREIGN GRAPHITE IN 1919. By A. H. REDFIELD. SECONDARY METALS IN 1919. By J. P. DUNLOP. GEMS AND PRECIOUS STONES IN 1919. By B. H. STODDARD.

CHROMITE IN 1919. By J. S. DILLER.

MINERAL WATERS IN 1919. By A. J. ELLIS.

CORRIGENDUM.—In the issue of February 28, p. 63 B, under Royal Photographic Society read "Dr. L. A. Levy" in lieu of "Dr. M. Levy."

## THE HABER PROCESS AT OPPAU.

J. R. PARTINGTON.

The synthetic ammonia plant of the Badische Company comprises two factories: the Oppau works, forming practically a continuation of the parent works at Leverkusen, and the Leuna works near Merseburg (Halle). Oppau, which is said to have cost £15,000,000, was commenced in 1913, with a capacity of 20 tons of ammonia per day; during the war this was increased to 200 tons. The synthetic nitrogen products made at Oppau in 1918 are stated to have been:—

	Tons per annum.	NH <sub>3</sub> equivalent.
Ammonium nitrate .. ..	10,000	4,250
Sodium nitrate .. ..	180,000	26,000
Nitric acid .. ..	40,000	10,800
Ammonia liquor* .. ..	—	40,000

\* Exported to Höchst for oxidation to nitric acid.

The Leuna works was erected since 1916, and has a capacity of 400 tons of ammonia daily. The combined output of Oppau and Leuna, 210,000 tons of ammonia per annum, is equivalent to over 800,000 tons of ammonium sulphate or 1,050,000 tons of sodium nitrate. Importation of the latter by Germany is no longer necessary. The German Minister of the Interior reported that Germany in 1916 made, by all processes, 400,000 tons of synthetic fixed nitrogen. Besides synthetic ammonia other salts, such as the chloride and nitrate, and mixed salts for fertilisers, are made at the Haber factories.

The power for both plants is derived from lignite. That for the Oppau works comes from the Ruhr district. The fuel is gasified in "Bamag" producers, 12 ft. by 25 ft., with rotary grates, a little steam being added to the air. In the Oppau works (to which the following description applies) the power plant generates 15,000—16,000 h.p. A row of 12 gas producers, each consuming 20 tons of lignite and yielding 2 million cb. ft. of gas per day, is built alongside a similar row of water-gas generators which provide the hydrogen. The latter use Ruhr oven-coke, are of the Pintsch type with rotary grates, 15 ft. by 25 ft., gasifying over 30 tons of coke to 3,000,000 cb. ft. of gas each daily. Some air is added, and the resulting gas contains about 40% H<sub>2</sub>, 30% CO, 12% CO<sub>2</sub>, and 18% N<sub>2</sub>. The water gas is treated by the Badische process (B.P. 26,770 of 1912; 27,117 of 1912; 124,760 of 1918; U.S.P. 1,115,776 of 1914; 1,200,805 of 1916), in which a mixture of water-gas and steam is passed over a catalyst consisting of ferric oxide, with promoters such as chromium oxide, at a temperature of 400—500° C., when the reaction  $\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{CO}_2 + \text{H}_2$  takes place. The catalyst remains active for two years. The catalyst plant comprises 24 units in two sections of two rows of 6 to 7 units. Each unit has two heat-exchangers and one elevated catalyst chamber; the latter is 16 ft. by 12 ft. by 10 ft. deep, with an oval cover carrying two 8-in. pipes, and contains two trays of catalyst. The heat-exchangers are 30 ft. by 15 ft. by 6 ft., and the whole apparatus is lagged, so that the reaction takes place without external heating. (The addition of a little oxygen or air to the gas has been described as a means of maintaining the temperature, a free flame burning in the catalyst chamber.) The gas issuing from the converters (in which the reaction is conducted at atmospheric pressure) contains a little carbon monoxide (according to Greenwood, "Industrial Gases," p. 163, this may be 2.5%), and must be purified from carbon dioxide, residual carbon monoxide, and hydrogen sulphide (from sulphur in the coke).

The purification (B.P. 120,546 of 1914; 9271 of 1914; 120,546 of 1918; F.P. 359,671 of 1916; U.S.P. 1,196,101 of 1916) is carried out by washing out the carbon dioxide with water under pressure (B.P. 11,878 of 1910; 124,761 of 1918); washing finally with soda (B.P. 15,053 of 1914) is not used. Carbon monoxide is taken out by washing with ammoniacal copper solution and hot caustic soda solution under high pressure (B.P. 1759 of 1912; U.S.P. 1,126,371 of 1915; 1,133,087 of 1915; J. Amer. Chem. Soc., 1921, 43, 1), which also takes out hydrogen sulphide. The copper solution contains excess of ammonia (not less than 6%) to prevent iron being attacked; the soda solution may contain 25% NaOH, and is brought in contact with the gas at 260° C. under 200 atm. pressure.

In the purification from carbon dioxide the gas, carried by a 3 ft. main from the converters, is compressed at 25 atm. and passed to the bases of eight steel towers, 30 ft. by 4 ft., packed with rings, into the tops of which water at 25 atm. pressure is injected by circulating pumps. The water issues charged with carbon dioxide; it is passed through Pelton wheels, in which 60 per cent. of the power is recovered, and the carbon dioxide evolved is collected. It is important that the amount of carbon dioxide recovered should be more than sufficient to convert all the ammonia produced into sulphate by the gypsum process, or into chloride by the ammonia-soda process. The gas is now passed through steel bottles which act as spray catchers to the high-pressure plant. Here it is brought to 200 atm. and delivered to the carbon-monoxide purifiers.

This plant consists of 16 towers of special steel, each in one piece, 25—30 ft. high by 2 ft. 6 in. external diameter, with flanged ends, and packed with Guttman balls. In the first eight of these an ammoniacal copper formate solution is circulated, in the remainder caustic soda ( $\text{CO} + \text{NaOH} = \text{H.COONa}$ ). The liquids are circulated by eight hydraulic pumps of forged steel, 200 h.p. total, and the ammoniacal copper solution, which absorbs ten times its volume of carbon monoxide, is passed down a 40 ft. tower, where the gas is released and collected. Spray traps in the form of vertical steel bottles are placed under these towers. The presence of 0.01 per cent. of carbon monoxide in the final gas is said to be injurious.

The purified hydrogen (with some nitrogen from the air mixed in the water-gas operation) is now passed to the catalyst plant by a high-pressure main. The nitrogen content is brought up to the ratio N<sub>2</sub>:3H<sub>2</sub> from a Linde plant, and the gas is dried.

The catalyst plant, in which the mixture is converted into ammonia, consists of fifteen steel bombs, each comprising two straight flanged sections, each 19.7 ft. long and 31.5 in. internal diameter, the walls being 7 in. thick. These are bolted together and the covers are held on by fifteen 4-in. studs. The walls are perforated at intervals of 1 ft. with  $\frac{1}{4}$ -in. tapering holes. Inside is a steel liner, making a gas-tight joint with the ends (U.S.P. 1,188,530 of 1916; D.R.P. 254,571 and 256,296 of 1911), and probably also a refractory lagging and a support for the catalyst, the internal diameter of the catalyst space being 19.75 in. The outer walls are heavily lagged, and reach 300—400° C.; the catalyst is at 600° C. The temperature is maintained by heat-exchangers, consisting of forged steel tubes, 19.7 ft. long and 15 in. external diameter, fitted internally with nests of  $\frac{1}{4}$ -in. steel tubes autogenously welded into end-plates. Each catalyst unit (costing 100,000 marks) is mounted vertically with its heat-exchanger inside a brickwork compartment with strong iron doors and planking, with a little of the bomb projecting above. Starting is effected by

adding oxygen (which does not come into contact with the catalyst) (D.R.P. 359,870 of 1911) or by electrical heating (U.S.P. 1,202,995 of 1916). An iron catalyst with a promoter (possibly molybdenum) is used (D.R.P. 265,295 of 1912; U.S.P. 1,188,530 of 1916).

The gas is circulated through the catalyst; argon and methane may accumulate up to several per cent.; 10 per cent. of the gas is lost by leakage.

The ammonia is removed from the gas by solution in water under pressure (D.R.P. 235,421 of 1908; 270,192 of 1912); liquefaction (U.S.P. 1,202,995 of 1916) is not economical. Three water-injector pumps are connected with nine sets of absorbers, each consisting of three water-cooled steel spirals set vertically, the upper spiral being 60 ft. above ground. The gas passes down the lowest spiral, rises to the top of the second spiral and passes down this; and similarly with the third spiral. Water flows down all the spirals by gravity. A solution containing 25%  $\text{NH}_3$  is obtained. The gas is dried and sent back to the catalyst system.

The personnel at Oppau comprises 1500 labourers, 3000 mechanics, 350 clerks and 350 chemists. The fuel requirements (McConnell, J. Ind. Eng. Chem., 1919, 11, 837) are 1750 tons of lignite (400 for gas engines, 1000 for steam for hydrogen plant and 350 for power plant) and 500 tons of coke (for hydrogen) per day. The daily cost of labour is £2300, of fuel £2300, and working expenses, interest, etc., £8700. The progress made since the laboratory experiments of 1906 to this immense factory in 1913—a period of only seven years—represents one of the greatest technical achievements of all time. Of particular interest is the association of the synthetic ammonia plant with the ammonia soda process.

The only other synthetic-ammonia factory outside Germany is the United States Nitrate Factory No. 1, at Sheffield, Alabama, erected during the war. This utilises the modified Haber process of the General Chemical Co. (U.S.P. 1,141,947—8 of 1915; B.P. 120,546 of 1918; 124,760—2 of 1918). The catalyst is prepared by impregnating pumice with nickel or ferric nitrate, heating to 550° C., reducing at this temperature with hydrogen, and treating with sodium and ammonia gas at 450° C. Sodamide is formed in the spongy metal, and the mass acts at 500° C. under 70 atm. (a much lower pressure than that used in Germany). The plant was designed to produce 21.5 tons of ammonia daily, and cost \$13,000,000; the synthetic ammonia section cost \$7,000,000, but requires another million for completion. Three units are installed. The plant was operated for a short time, but great difficulties were encountered owing to faulty design, and the factory was closed in the beginning of 1919, about three months after work began. The process developed into a straight blue water-gas make, followed by addition of air in the hydrogen converters for flame combustion to maintain the temperature. Caustic scrubbing was discarded as giving too much sediment and ammoniacal cuprous carbonate alone used at low temperatures. The catalyst furnaces, which were operated at 100 atm., were similar to the well-known Badische converters for sulphur trioxide, but much thicker and capable of withstanding pressure. The catalyst was contained in the internal battery of iron tubes. Self-heating was not attained (R. S. Tour, J. Ind. Eng. Chem., 1920, 12, 844).

**APPOINTMENT OF COMMITTEE UNDER THE DANGEROUS DRUGS ACT.**—The Home Secretary has appointed a committee of five to consider objections to, and to advise concerning possible modification of, the draft Regulations issued under the Dangerous Drugs Act.

## THE MANUFACTURE OF HAND-MADE FILTER PAPER.

J. BARCHAM GREEN.

The following brief notes on the manufacture of hand-made filter paper will be of interest in view of the circumstances that the manufacture was practically initiated in this country during the war, and that English papers have now to a great extent replaced the foreign-made papers which were previously in general use.

The foundation of a good paper is suitable rag; old muslin curtains have been found to give the best results for fast filtration, but where strength is required a strong rag, like best canvas or old linen shirts, is often added. After the rags have been sorted and cut by hand the dust is shaken out in a rotary machine made of wire gauze, then buttons and other foreign matter are picked out, and the rag is hoiled with alkali for 1 to 4 hours. Opinions differ as to whether alkali should be used, but 2 to 3 lb. per cwt. of dry rag should not be harmful if the hoiling is done without high pressure. The hoiled rag is reduced to pulp in water in a beater provided with bronze knives fitted on to a solid drum, which can be lowered until the knives touch a grooved bronze plate. Paper that filters quickly is obtained by reducing the proportion of rag to water and increasing the speed of the beating, but much depends on the skill with which the roll or cylinder is adjusted to the grooved plate; when the beating has been done very quickly the finished sheet will sometimes show the threads of the muslin rag used. The pulp is usually bleached with chloride of lime, the excess of which must be completely washed out with pure water. After beating, the pulp is strained to remove knots and lumps, run over magnetic separators to remove any particles of iron that may be present, mixed with water, and then run into a vat, either of stone or of iron lined with lead. A layer of pulp is obtained by dipping a mould covered with wire gauze into the vat, removing it and allowing the water to run off; the layer is pressed on to a felt and a pile of alternate layers of pulp and felt is put under a hydraulic press. The pressure applied largely determines the texture of the paper; if it be too great a hard paper that filters slowly will be produced. The paper is dried either on a steam-heated cylinder or by hot air, sorted, and stocked as sheets or circles. Circles are cut from a wad of about 100 sheets by means of a steel cutter, and when large circles of, say, 50 cm. diameter are cut from a sheet of 50 cm. square, the corners are wasted if they cannot be sold. This explains why the price of circles is apparently out of proportion to their actual size.

In the manufacture of filter paper particular attention has to be paid to its purity, strength, filtering speed, and ash content. To obtain a pure paper neutral spring water, free from organic matter and containing little calcium bicarbonate, must be used. When testing for strength the paper must be wet; the strength when dry is often very different from the strength when wet, especially in the case of very strong papers intended for use with a filter pump.

Roughly speaking, the stronger a paper the more slowly it filters, and to obviate this difficulty the manufacturer selects a very strong fibre, such as new linen, and beats it very rapidly to a pulp; otherwise it becomes slimy (if beaten long enough it would be converted into hydrocellulose and produce a sheet of parchment which would be waterproof). Strength can also be obtained by using thicker paper, but as this filters slowly some makers produce a paper with a crinkled surface. Such a surface cannot be given to hand-made papers, but a better result is obtained by folding the paper fan-

wise to produce a fluted surface. The speed of filtration is ascertained by finding the time required for pure water to pass through the paper at a definite temperature. Liquids containing suspended solids, such as barium sulphate, are less suitable, as mistakes may be made in preparing them; they must, however, be used in testing the clarity of the filtrate.

Before the war certain foreign firms sold "ashless" papers having a certain ash content, and their figures were usually taken as correct; but when the manufacture was started in this country it was found that the ash content of these papers varied very greatly and that it was seldom as low as the figure stated. English papers, as now made, usually contain a little less ash than is indicated on the wrapping. English manufacturers now produce a large variety of papers among which types can be found suitable for many special purposes; and the makers are usually prepared to satisfy individual requirements. Complaints of bad quality often arise from the use of papers for purposes for which they are unsuited; they could in most cases be avoided by careful reference to the manufacturer's catalogue before purchasing.

## THE MANUFACTURE OF SULPHURIC ACID IN THE UNITED STATES.

Bulletin 184, recently issued by the U.S. Bureau of Mines, contains a considerable amount of information relating to the manufacture of sulphuric acid in the States. In 1865 only some 37,500 tons of acid, calculated as 100 per cent., was manufactured; in 1880 this had become 265,630 tons, and in 1900 over one million tons. The table which follows gives the production for the last six years before the armistice. It should be noted that all quantities have been calculated into the equivalent of 100 per cent. acid, as in the report different ways of stating the strength are given:—

Year.	Concentrated acid produced as 66° B.		Total production as strengths calculated to 100% H <sub>2</sub> SO <sub>4</sub> .	
	Tons.		Tons.	
1913 .. ..	810,000	..	2,239,930	..
1914 .. ..	720,000	..	2,280,000	..
1915 .. ..	1,160,000	..	3,173,800	..
1916 .. ..	1,930,000	..	3,938,000	..
1917 .. ..	2,100,000	..	4,505,000	..
1918 .. ..	2,400,000	..	4,661,300	..

Towards the end of 1918 the producing capacity was approximately 6 million tons, about 40 per cent. of the total being contact acid. Government and explosive manufacturers' plants accounted for about 22 per cent. of the total production, the rest being fertiliser and general chemical works plants. When the armistice was signed work was stopped at once on plant in course of erection designed to produce some 450,000 tons per annum.

The distribution of the factories is described by means of a map, tables and a list of the individual works, the greater number being in the Eastern and South-eastern States.

The proportion in which the various industries were using acid in 1918 is shown below:—

	Per cent.
Explosives (military) .. .. .	33.5
do. (domestic) .. .. .	2.5
Fertilisers (superphosphates) .. .. .	28.4
Oil refineries .. .. .	3.6
Chemicals, drugs, and ammonium sulphate .. .. .	9.9
Steel pickling and galvanising .. .. .	9.3
Fabrics, tanning, etc. .. .. .	1.3
Paints, lithopone, glue, etc. .. .. .	1.4
Metallurgical, including storage batteries .. .. .	3.9
Miscellaneous .. .. .	1.0
	100.0

The normal requirements of the United States are estimated to be equivalent to about three and a half million tons of 100 per cent. acid, of which about one-half is required for superphosphates, so that a considerable margin of producing capacity exists.

The influence of the war on the proportions of the various sources of sulphur as raw material is well illustrated in the next table:—

	Percentage of total quantity.		
	1914.	1917.	1918.
Brimstone .. .. .	2.6	32.6	48.0
Pyrites (Spanish) .. .. .	50.0	22.9	7.6
do. (domestic, including coal brasses and pyrrhotite) .. .. .	15.3	11.8	12.7
do. (Canadian) .. .. .	7.9	6.9	7.5
Zinc ores .. .. .	13.2	18.1	16.1
Waste gas from copper smelters .. .. .	10.5	7.7	8.1
	100.0	100.0	100.0

Practically all the brimstone was obtained by the Frasch process of mining from the coastal plain deposits of Louisiana and Texas. Owing to the ease of handling and to the cleaner gas obtained, brimstone is considered to be worth 3 to 4 cents more per unit than a 40 per cent. pyrites if the acid maker gets nothing for his burnt ore. The price of Spanish pyrites rose during the war to as much as 30—35 cents per unit, but it has since fallen again, the quotation in July last being 16 cents. Much attention has been given to the recovery of pyrites from waste coal, and it is concluded that the coal mines of the Eastern States could produce over 1½ million tons of pyrites per year with an average content of 40% sulphur.

All the zinc works of Illinois, Indiana, Ohio, Pennsylvania, and West Virginia are producing acid, but those of Kansas, Missouri, and Oklahoma have not yet done so. At the copper works some very large plants exist, that of the Tennessee Copper Co. being capable of producing acid equivalent to over 625 tons (of 100%) per day.

Both the chamber and the contact systems are described at considerable length, and the following are the chief points noticeable about the American practice:—Owing to the large use of brimstone, special devices for burning this material have been developed, among which may be mentioned the Tromblee and Paul rotary and the Vesuvius vertical burners; Herreshoff and Wedge types of mechanical fine pyrites roasters seem to be most in favour, and Hegeler, Spirlet, and Wedge (muffled) are in use for zinc ores. The Cottrell electrical precipitator has given satisfaction both as a dust eliminator for burner gas and as a condenser for fumes from acid-concentration plants. Towers constructed of acid-proof brick and cement without a lead casing are in use, and steel framing is replacing wood as a material for supporting the chambers. Much attention appears to be given to intermediate towers of various types, and a full description is given of the "Anaconda Packed Cell" system, in which the chambers are replaced by towers packed with special tiles.

In metallurgical and other works where the composition of the burner gas is likely to vary the Fairlie system of analytical control is advocated. Concentration is effected in cascades, Kessler plants, and in tower systems of local design, such as the Kalbpery and the Chemico Concentrators, but no mention appears to be made of the Gaillard system. The Mannheim, Badische, Grillo-Schroeder, and Tentelew contact systems are all in use and are described and compared, as are also the various types of gas-cooling towers, dust filters, drying and absorbing systems, and the methods of preparing and revivifying the catalysts.

Comparisons of pre-war and post-war costs of plant and acid for both chamber and contact systems are given, and the report concludes with a short account of the methods of utilising burnt pyrites and nitre cake.

## SOCIETY OF CHEMICAL INDUSTRY.

### ANNUAL MEETING, 1921.

With reference to the cancelled sailing of the s.s. "Megantic" from Liverpool to Montreal on August 13, it has been decided to postpone for another month the choice of the steamship to convey the presidential party.

The following communication has been received from the Committee of the Montreal Section:—

Plans are rapidly maturing for the Annual Meeting of the Society to be held in the latter part of August. A strong committee of the Montreal Section is arranging an elaborate programme of great interest to visiting members, details of which will be announced later. The Windsor Hotel, situated in the upper heights of the city of Montreal, one of the beauty spots of the Canadian metropolis, has been chosen as headquarters. A unique feature of Montreal is the glimpses it affords of the old French régime side by side with examples of the modern spirit of commercialism, e.g., the historic Bonsecours Church with its immense statue overlooking one of the world's most modern harbours, where huge grain elevators give evidence of one of the main sources of the country's wealth. Plans are being made for an extended trip in Canada which will provide opportunities to inspect typical Canadian manufactures and to view the primitive beauty of forest and stream. As the steamships plying between England and Montreal touch at Quebec, a few enjoyable hours may be spent in rambling round the ancient capital and its environs so rich in historic interest. It is considered none too early for members to begin making their plans with a view to attending what should prove to be a particularly interesting meeting.

### MARCH MEETING OF COUNCIL.

At the monthly meeting held on March 11, the President moved a vote of condolence to the family of the late Lord Moulton, which was carried unanimously, and the Secretary was asked to attend the memorial service. Sir William Pope intimated that the nomination of the president for 1921-22 would be considered at the next meeting, also the nomination of vice-presidents and the election of hon. treasurer and hon. foreign secretary. Thirty-nine new members were elected (21 home, 4 colonial, 3 Indian, and 11 foreign), and Mr. John Spiller, an original member who has done valuable work for the Society over a long period of years, was unanimously elected an honorary life member.

The Hon. Treasurer reported that the sum of £13,124 had been received as proceeds of the realisation of the non-trustee securities of the late Dr. Messel's bequest, and it was resolved to invest the whole of this sum in 5 per cent. War Loan, 1929-47. It was also stated that the Finance Committee would in due course prepare a statement showing how the legacy had been dealt with. A separate account will be kept of the monies derived from this bequest.

On the report of the Publications Committee, it was decided to authorise the reprinting and circulation of approved articles from the Review bearing upon public policy, as occasion required and due regard being paid to expense; and it was agreed that the Council, through the Government and Parliamentary Committee, should associate itself with the Pharmaceutical Society and other interested bodies in opposing the regulations recently issued under the Dangerous Drugs Act. The report of the Edinburgh and East of Scotland Section was presented to the Council.

## CHEMICAL ENGINEERING GROUP.

A conference on "Waste Waters" was held at the Technical College, Huddersfield, on February 24. Dr. H. H. Hodgson, who presided in the absence, through illness, of Mr. J. A. Reavell, commented upon the importance of the work done by the Society and the Group in spreading useful knowledge applicable to the development of industry, and complimented the Group upon its enterprise in holding its meetings in various parts of the country instead of in one locality, as was the practice of many societies. He then called upon Mr. E. V. Chambers to read his paper on "Plant for the Purification of Industrial Waste Waters, with Recovery of Useful By-products."

The woollen industry yields three classes of effluent—that from washing the raw wool, that from scouring yarn and pieces, and the effluent from the dyeing process. In each case the effluent is passed through a rotating circular screen to remove fibre, and the subsequent treatment, which is partly mechanical and partly physical, varies with the nature of the liquid to be purified. Suspended solids are eliminated in settling tanks by gravity or centrifugal filters, and colloidal matter is similarly removed after pre-treatment of the liquor with alumino-ferrie or other precipitant. The author then described—first generally, then in detail—the process for purifying the effluent from a woollen piece-scouring factory, which consists, briefly, in removing the grease by saponification with alkali, and then treating the liquor for recovery of soap, soda, and oil. A typical plant with a capacity of 22,000 galls. a day of strong scouring liquor was described and illustrated. The next portion of the paper dealt with the treatment of general effluent to produce a non-polluting discharge into a stream. In its broad outlines the scheme is the same as that mentioned above, but additional factors have to be taken into account, and labour charges must be cut down to a minimum. A good example of the feasibility of conducting effluent treatment as a source of profit is shown by the recovery of wool grease, lanolin, and potash from wool scourings; it is estimated that nearly 50,000 tons per annum of wool fat and about 20,000 tons of potash (reckoned as carbonate) are available for recovery in this country. The successful Smith-Leach process was described and also the modern centrifugal method which yields wool fat of twice the value and at half the cost of the "acid" or chemical process. Details were given and photographs and diagrams shown of a plant for carrying out the centrifugal process. The treatment of the pickling liquor obtained in various metal-working factories, e.g., wire-drawing works, where it contains about 0.5 per cent. by weight of free hydrochloric acid and about 20 per cent. of ferrous chloride, consists in adding enough sulphuric acid to liberate all the combined chlorine as hydrochloric acid and in obtaining this by distillation, the iron being recovered as ferrous sulphate. The operation of a typical plant was described and explained.

Mr. O. Reynard, of Bradford, said that he had found that the centrifugal method of extracting wool fat was very successful from the qualitative standpoint, but that he had only been able to recover about 40 per cent. of the fat instead of 60-75 per cent., as mentioned by the author. The difficulty probably lay in the existence in the emulsion of the wool fat in two states of "fineness"; the coarser was rapidly removed and represented the 40 per cent. extraction, but the finer could not be completely separated. Mr. Chambers, in his reply to Mr. Reynard and others, said that the results given in his paper had mostly been obtained on a commercial scale at works in Huddersfield. The "frothing" process had been tried but had invariably led to bacterial decomposition of the wool



fat unless the air had been sterilised or filtered before use. The "churn" process had also been experimented with, but it was only partially successful and far less effective than the methods described in the paper. No trouble had yet been experienced from the presence of arsenic in the acid recovered from waste pickling liquor (*cf. s.*), as the design of the plant obviated this.

The full report of the meeting will be published in Volume III of the *Proceedings* of the Group.

#### NEWS FROM THE SECTIONS.

##### TORONTO.

"The Story of Petroleum" was the subject of an address delivered by Mr. W. A. P. Schorman, of the British-American Oil Co., on January 28. The chairman of the Section, Mr. M. L. Davies, of the Standard Chemical Co., presided, and there was a very large attendance of members and visitors. The address was illustrated by kinematograph pictures showing the activities of the oil industry from the drilling and shooting of the oil wells to the distillation processes in the refineries. Referring to the recent discovery of oil at Fort Norman in North-West Canada, Mr. Schorman said that he believed that oil had been found there in paying quantities, but actual figures were not yet available. The oil that had been found would flow freely at a temperature well below  $-60^{\circ}$  F. and did not solidify above  $-90^{\circ}$  F. This was a very valuable characteristic of the oil, for the temperature in that territory often reached  $-60^{\circ}$  F. during the winter. Another very valuable circumstance was that the oil yielded over 50 per cent. of the lighter hydrocarbons, so that when the territory was sufficiently developed a large supply of gasoline should be secured. The discovery of the oilfield was made by a Canadian geologist, Mr. Boswell. Mr. C. B. Parsons, president of the British-American Oil Co., spoke optimistically of the development of the oil industry in Canada, and expressed his belief that oil would yet be found in other more accessible parts of the Dominion. The need for developing an oil industry in Canada was very great.

##### OTTAWA.

On December 16, 1920, about 40 members of the Section assembled to see a kinematograph film entitled "The Story of Coal," kindly lent by the Sullivan Machinery Co., of Chicago. The film showed the mining of coal in one of the model mines in Pennsylvania and many views of plant used in manufacturing mining machinery.

At a meeting in the Carnegie Library, Ottawa, on January 13, Mr. E. Stansfield presiding, a discussion arose on the Bill to be introduced in the Ontario Legislature dealing with the legal status of the engineering profession, and it was decided to convene a meeting of the chemists of Ottawa to discuss the subject more fully. Dr. R. S. Stevens then gave an address on "Vitamins" in which he traced the history of the development of this branch of biochemistry and summarised our present knowledge of the subject. Colonel J. T. Janson followed with an address on the more chemical aspects of these food factors. Sixty-five members and guests were present at this meeting, including medical men and representatives from the University Women's Club.

A joint meeting was held with the Engineering Institute of Canada on February 10, the chair being taken successively by Lt.-Com. C. P. Edwards, chairman of the Ottawa Branch of the Engineering Institute, and Mr. E. Stansfield, chairman of the Section. The attendance numbered 65. Capt. E. M. Dawson read a paper in which he reviewed the cement industry and described recent

work on super-cements. He outlined the processes involved in the manufacture of Portland cement, dealt more in detail with the methods of testing raw materials and products, and finally reviewed the improved methods of making cement involving the use of what he termed "cata-cell," a substance which is introduced into all super-cements. He claimed that super-cement is superior in every way to Portland cement, because it is waterproof and more durable, owing to the fact that a "cata-cell" promotes more complete hydration. A long discussion followed.

##### LONDON.

A meeting of the London Section was held at Burlington House on March 7, Mr. Julian L. Baker presiding. Two papers were presented, the first of which was by Dr. J. C. Drummond on "Factors Influencing the Food Value of Lard and Lard Substitutes." It has often been stated that animal fats contain the fat-soluble vitamin A, and that vegetable fats are deficient in this substance; no such general rule, however, can be laid down since it has not hitherto been identified in lard. Two causes contribute to the low vitamin content of lard:—(1) The diet usually given to pigs in this country is often deficient in vitamin A; and (2) during the process of lard manufacture much of the vitamin, if present, is destroyed owing to exposure of the fat to oxygen at high temperature. If pigs are fed with a material rich in vitamin, such as grass or cod-liver oil, not only do they grow much better, but their fat contains plenty of this important factor. It would appear that the destruction of the vitamin is due less to the high temperature to which the fat is subjected in the subsequent refinement of the lard than to the action of atmospheric oxygen.

The second paper, entitled "The Stability of Benzoyl Peroxide," was read by Dr. R. C. Farmer. Benzoyl peroxide is of industrial interest as an oxidising agent, and has been used for several purposes under the name of "Lucidol." It is a powerful antiseptic, and has been used for the treatment of burns and ulcers. It acts as a drier for linseed and other drying oils, and has been used to a small extent as an explosive. The substance is unstable whether it be crude or pure, but immunity against explosion can be attained by mixing it in the wet condition with excess of salt and then drying.

##### NEWCASTLE.

At a joint meeting of the Section with the Cleveland Institution of Engineers, held at Middlesbrough on March 7, Mr. C. F. Tidman, in a paper on "Alcohol from Coke-oven Gas," described further experiments which had been carried out at Skinningrove on the absorption of ethylene by sulphuric acid and its conversion into alcohol.

In a brief historical introduction it was stated that Hennell had published papers in 1827-28 on the production of alcohol from ethylene, and that he therefore deserved to rank with Wöhler as the first to produce an organic compound from inorganic constituents. A series of laboratory experiments was described which showed that the ethylene was most rapidly absorbed by sulphuric acid of 95.4% at about  $80^{\circ}$  C. with turbulent contact; and it was deduced that nine towers, each 10 ft. diam. by 80 ft. high, would be required for the absorption of the ethylene contained in the coke-oven gas at Skinningrove, where 800 tons of coal are carbonised daily; of these nine towers the volume of six would be occupied by packing material. In further laboratory experiments it was observed that when ethylene and sulphuric acid are shaken in a flask, the addition of silica increases the absorption by about 5 per cent.; and it was concluded that whether



absorption towers or mechanical washers be adopted for large-scale use, it should be possible to remove 70–80 per cent. of ethylene from the coke-oven gas by allowing a contact of 2½–3 minutes between it and the sulphuric acid. Mr. E. Bury found that 18 per cent. of the sulphuric acid put into circulation was available for ethylene-absorption, i.e., that a 95% acid would continue to absorb until its concentration was reduced to 77%; further work has shown that the absorption can be carried still further when a mechanical agitator is used. The great advantage of a mechanical washer over a tower-scrubber system is the much smaller size of the plant required.

The recovery of the alcohol from the reaction mixture was found to be best effected by distillation with steam under reduced pressure, the bulk of the alcohol being obtained as a 15% concentrate. After rectifying up to 87%, it gave good results when used as motor fuel. The alcohol obtained by this method contains various impurities, including diethylene disulphide and a yellow oil of unknown composition, the former of which is due to the action of the hydrogen sulphide present in the coke-oven gas, and a method of eliminating it has been worked out which consists in treating the coke-oven gas with a large excess of sulphur-dioxide solution on the counter-current system, the excess of dioxide being removed by subsequent scrubbing with water and used over again. That pure alcohol can be made from coke-oven gas is certain, but it has yet to be done on a commercial scale. By using purified coke-oven gas and alcohol recovered by distillation under reduced pressure, it should be possible to obtain an 80% conversion of the absorbed ethylene, and by using an acid stronger than 95% it should be possible to obtain a higher ethylene absorption than 70%.

Prior to the meeting, at which over 150 were present, a visit was made to the By-product Works of Messrs. Sadler and Co., Ltd., Middlesbrough.

#### EDINBURGH AND EAST OF SCOTLAND.

The hon. local secretary's report on the work of the session, which was read at the annual meeting held on March 8, records the holding of six meetings attended by an average of forty members. Twenty new members joined the Section, twelve were lost by deaths and resignations, and ten by transference to other Sections; the total membership to date is 157. The Committee has now lost the able services of the chairman, Dr. D. S. Jordan, who is leaving to take up an appointment in Brussels, and Dr. A. C. Cumming is also leaving Edinburgh to engage in chemical manufacturing work in Liverpool. The thanks of the Committee are accorded to the Council of the North British Branch of the Pharmaceutical Society, and to its secretary, Mr. J. Rutherford Hill, for allowing the Section to use the Society's hall for its meetings.

#### BIRMINGHAM.

A paper on "Colour in Water: Its Nature and Removal" was read by Mr. Joseph Race at a meeting held on March 10 at Birmingham University. The paper was confined to a consideration of brown peaty waters which are found in various countries. The properties of these waters were fully described, and a résumé was given of the evidence of the colloidal nature of the colouring matter, together with details of experiments illustrating the practical importance of the behaviour of the colloids on the addition of an electrolyte, such as alum. It was stated that the effect of alum on coloured waters had been intensively studied in America, because the colour in many waters in that country is often very high and the water consumption is four to five times that in England. The effect of these causes

on filter design was indicated. The study of alum flocculation has led to the adoption of the "excess alum" method, and this has resulted in considerable economies being effected at many plants.

Removal of colour by electrolytic methods was also discussed and a detailed description was given of an experimental plant in which aluminium electrodes were employed. The results showed that when precipitation is effected in this manner the amount of aluminium consumed is very much less than when alum is used; also that the alkalinity, and consequently the carbonic acid content, remain unchanged. The costs, calculated on a run of 20,000 gallons, were given, together with analyses of the various precipitates obtained with both aluminium and iron electrodes.

An informal dinner for members of the Section—the first since 1913—was held in the White Horse Hotel on March 14, and was attended by about 30 members.

#### MANCHESTER.

The annual supper of the Section was held on March 10 in the Albion Hotel, Manchester. Mr. John Allen presided, and about 130 members and friends were present. On the announcement of the death of Lord Moulton all present rose in silence to express their regret. An informal musical programme occupied a portion of the evening.

### MEETINGS OF OTHER SOCIETIES.

#### INSTITUTE OF METALS.

The Spring Meeting of the Institute of Metals, held at the Institution of Mechanical Engineers on March 9 and 10, was presided over by Sir George Goodwin and was well attended. The subject of season-cracking in brass was brought forward in a paper by Messrs. H. Moore, S. Beckinsale and Miss Mallinson, who produced evidence, from the examination of a very large number of cold-worked articles at Woolwich, in favour of the view that the cracking is caused by chemical corrosion, especially by traces of ammonia, acting selectively on the intercrystalline material. The presence of initial stress is essential to the development of season-cracks, and the composition of the alloy is of secondary importance. The discussion showed that cracking may sometimes occur in new rods, especially in winter, under conditions which seem to exclude the possibility of corrosion, and it was also pointed out that the phenomena of fire-cracking and season-cracking are essentially similar, although the former are almost certainly not of chemical origin. Fortunately, the liability to crack is removed by low-temperature annealing, which is without any softening effect on the metal. As is usual when this subject arises, a part of the discussion resolved itself into a duel between the defenders and the opponents of the hypothesis of an amorphous intercrystalline cement. The whole subject is to be considered at the forthcoming joint meeting arranged by the Faraday Society. The same authors also dealt with another form of intercrystalline brittleness, produced in copper by the action of reducing gases. This action is due entirely to the presence of cuprous oxide, and copper which has been thoroughly deoxidised is immune. The addition of a small quantity of zinc is particularly recommended for this purpose, and attention is called to the useful properties of an alloy of copper containing from 2.5 to 3% of zinc, which works well hot or cold, and will stand severe deformation. It casts well, and is not subject to season-cracking.

Prof. C. A. Edwards and Mr. A. M. Herbert described measurements of the dynamic hardness of copper-zinc and copper-aluminium alloys at high temperatures, showing that critical temperatures in such alloys are more readily revealed by dynamic testing than by the slow application of a tensile stress. An interesting point mentioned in the discussion was that workmen when heating billets of such alloys determine their approximate temperature by poking with a pointed rod, a rough application of a dynamic hardness test. Mr. P. H. Brace, of Pittsburgh, communicated an account of the metallurgy of calcium, describing the modern form of electrolytic cell, using pure calcium chloride as the electrolyte, and tabulating the principal properties of the metal. It is possible that calcium may find more extended uses in metallurgy. In the absence of Prof. H. C. H. Carpenter, through illness, the further paper on the recrystallisation and crystal growth in metals, in collaboration with Miss C. F. Elam, was read by the latter. The new results deal mainly with aluminium, and it is shown that the complete destruction of the crystal structure, often described by previous workers as resulting from severe cold-working, does not take place, and that the confused micro-structures so often recorded are due merely to the difficulties of etching this material. The birth of new crystals in the metal during annealing is clearly shown, and a comparison with brass and with aluminium-zinc alloys proves that the new crystals arise in the boundaries of the old. The remaining paper, by Dr. J. L. Haughton, continued his earlier work on the revision of the copper-tin system, and included a diagram covering the range from 35 to 100 per cent. of tin. Even now, some of the transformations are difficult to understand, and the author's careful determinations of electrical resistance at varying temperatures indicate changes of which there is at present no explanation.

#### THE CHEMICAL SOCIETY.

The Annual General Meeting was held at Burlington House on March 17, when the following elections were announced by Sir J. J. Dobbie:—*President*: Sir James Walker; *Vice-Presidents*: Prof. H. E. Armstrong, Sir J. J. Dobbie, Prof. W. H. Perkin, Sir W. J. Pope, Dr. A. Scott, Sir W. Tilden, Prof. F. G. Hopkins, F. S. Kipping and J. F. Thorpe. *Ordinary Members of Council*: Prof. J. S. S. Brame, C. H. Desch, T. S. Patterson, Drs. T. Slater Price, R. Robinson, N. V. Sidgwick, and Messrs. E. V. Evans, H. B. Hartley, and W. Rintoul. The Longstaff Medal was presented to Prof. J. F. Thorpe, and Sir J. J. Dobbie read in abstract his presidential address on the value of the study of the history of chemistry.

In the evening the Anniversary Dinner was held at the Hotel Cecil, Strand, W.C. 2. Among the guests were the Right Hon. Sir Alfred Mond, the Right Hon. Lord Bledisloe, Prof. C. Meunier (vice-president of the Société Chimique de France), Sir J. Dewar, Sir W. Tilden, Prof. C. S. Sherrington, and the Hon. Mr. Justice Sargant. The number present was 224.

After the loyal toasts Sir Alfred Mond, in proposing the Chemical Society, referred to its distinguished past presidents, and expressed the fear that the spirit which drove Hofmann from England and which helped to drive the dye industry to Germany was not yet extinct. "Science and research are not the fads of the few or the follies of the aged, but vital necessities to the existence of the nation." The exhaustion which followed the Napoleonic wars was greater than that which existed today, and the key to recovery lay in some great improvement in our methods of production, some great discovery as fundamental and far-reaching as that of the steam engine in its epoch.

Sir J. J. Dobbie, in reply, outlined the more important changes and developments which had occurred in the Society during the past two years. In particular, he emphasised the reformation of the charter on a more democratic basis, the admission of women to the fellowship, and the financial stringency. The number of fellows had increased from 3205 in 1914 to 3569 in 1919, and last year there were 3721; but more important than this growth in membership was the recent increase in the number of papers printed in the Transactions; from 272 papers in 1914 the number had fallen to 89 in 1918, but last year it rose to 182. The insufficient accommodation afforded by the Society's rooms in Burlington House was then referred to, and the hope expressed that means would be found to realise the aspirations of chemists as embodied in the proposals of the Federal Council for Pure and Applied Chemistry.

The toast of the Past-presidents, coupled with the names of Sir James Dewar, Sir Edward Thorpe, and Sir William Tilden, was given by Prof. H. B. Dixon, who outlined in brief but witty terms the outstanding achievements of these three guests of honour, and in their acknowledgments, Sir J. Dewar and Sir W. Tilden gave interesting reminiscences of their early associations with some of the chemical pioneers of the last century. Sir E. Thorpe was, unfortunately, absent owing to illness.

Prof. F. G. Donnan gave the toast of the Guests, which was replied to by Prof. Moureu, the Hon. Mr. Justice Sargant, and Prof. C. S. Sherrington.

#### THE OPTICAL SOCIETY.

At a meeting of the Optical Society held at the Imperial College on March 10, Mr. T. F. Connolly described and exhibited "A Handy Form of Measuring Microscope." The instrument comprises a microscope system of relatively low power; a double image prism traverses the object space, and is controlled by the rotation of a milled hand operating a spiral slot. Exit pupil discs of telescopes or other optical instruments and Brinell impressions can be measured by a single contact setting of the edges of the duplicated discs. The range in the experimental instrument is from 1 to 6 mm., and the accuracy when used with a fixed focus for Brinell impressions is about  $\pm 0.02$  mm. The reading scale is very open and is divided to 0.1 mm. No vernier is used.

#### PERSONALIA.

Dr. F. H. Hatch has been appointed adviser to the Mines Department on questions relating to the metalliferous mining industry.

The Albert Medal of the Royal Society of Arts for 1920 has been presented to Prof. A. A. Michelson, professor of physics in the University of Chicago.

Dr. F. C. Thompson, Sorby Research Fellow in the University of Sheffield, has been appointed to the chair of metallurgy in the University of Manchester.

Prof. J. S. S. Brame, professor of chemistry in the Royal Naval College, Greenwich, has been elected president of the Institution of Petroleum Technologists for 1921-22.

Sir Edward Thorpe (Great Britain), Prof. Le Chatelier (France), Prof. Ciamician (Italy), and Dr. Ernest Solvay (Belgium) have been elected honorary foreign members of the Chemists' Club, New York.

The principalship of the Manchester Municipal College of Technology has been filled by the appointment of Prof. B. Mouat Jones, who has been professor of chemistry in the University of Aberystwith since 1919.

## NEWS AND NOTES.

## JAPAN.

**The Wood-Distillation Industry.**—The wood-distillation industry in Japan originated during the war, when the rubber planters in the East were entirely cut off from the German sources of supply of acetic acid, and Japan was called upon to meet their entire requirements. The consumption of calcium acetate in Japan has been large but variable during the last eight years; in 1913 it was 4049 long tons, in 1916, 577 t., and in 1919, 5398 t. Imports, formerly derived mainly from England and the United States, have since 1916 come from the latter country only. The demand for acetone is estimated at 600–700 tons per annum, and the country can now supply its own requirements. Much progress was made in the production of methyl alcohol until 1917; but since that year it has diminished considerably, partly owing to increased importation from America. The chief product exported is acetic acid, which goes to the Straits Settlements, British India, and the Dutch East Indies. Exports of this acid were 669 t. in 1915, 2470.6 t. in 1917, 2137.8 t. in 1919, and 890 t. during the first four months of 1920. The export trade diminished in 1920.—(*Chem. Ind.*, Dec. 29, 1920.)

**Menthol and Peppermint Oil in Japan.**—Menthol is obtained as a residue in the refining of the oil distilled from a variety of peppermint grown in the Okayama and Hiroshima prefectures, in Yamagata, and in Hokkaido (the chief source). The crystals are exported mainly to America and the peppermint oil to England (formerly Germany). The total production, average prices, and exports during the period 1909–1920 were as follows:—

Menthol.				Peppermint Oil.				Exports from Yokohama.
Produce.	Average price.	Total exports.	Produce.	Average price.	Total exports.	Produce.	Average price.	
Long tons.	Sh. per lb.	Long tons.	Long tons.	Sh. per lb.	Long tons.	Long tons.	Sh. per lb.	Long tons.
1909 ..	—	7.35	60.35	—	4.7	—	—	—
1910 ..	—	8.8	31.74	—	5.1	—	—	—
1911 ..	—	15.5	66.97	—	6.4	—	—	41.14
1912 ..	—	21.2	73.48	—	5.6	—	—	39.79
1913 ..	—	19.3	137.26	—	6.1	—	—	75.55
1914 ..	142.9	9.2	158.67	145.8	3.3	—	—	92.87
1915 ..	223.9	8.5	188.76	257.0	2.6	—	—	75.80
1916 ..	210.5	10.1	227.73	241.6	3.1	—	—	104.13
1917 ..	252.8	10.0	147.92	254.2	2.8	—	—	68.26
1918 ..	196.5	14.4	132.32	232.4	3.4	—	—	68.46
1919 ..	147.3*	31.6	132.59	89.3*	7.2	—	—	85.95
1920 ..	118.3*	43.0†	7.95†	118.3*	10.2†	—	—	11.36†

\* Unofficial estimate.

† Jan.-June, 1920.

—(*U.S. Com. Rep.*, Feb. 3, 1921.)

## UNITED STATES.

**Magnesium in 1919.**—A report of the United States Geological Survey, issued in 1920, states that 127,465 lb. of metallic magnesium, valued at \$247,302, was made by three companies in 1919, a decrease of 55 per cent. in quantity and 60 per cent in value compared with 1918. The decrease was due to the cessation of the demand for war purposes and to lack of foreign orders. Most of the magnesium marketed was in stick form, but over one-tenth of the output was sold as powder. Average prices were \$1.83 per lb. for stick magnesium and \$2.85 for the powdered metal. Magnesium is prepared from anhydrous sodium-magnesium chloride, left after the extraction of salt from brines at Midland, Mich., and Pomeroy, Ohio; from magnesium chloride from the waters of the Great Salt Lake, Utah, and from sea water at San Francisco Bay; from Californian magnesite; and from precipitated magnesium carbonate. Metallic magnesium is mainly used as a deoxidising agent in metallurgy, but its use as a constituent of alloys is increasing and may eventually become the more important.

**Abrasive Materials in 1918.**—According to "Mineral Resources of the United States" (1918, Pt. II., pp. 1171–87), the total value of all abrasive materials, exclusive of felspar and quartz, consumed in the United States in 1918 was \$10,991,964, of which about 26 per cent. represented natural abrasives. Imported abrasives were valued at \$1,187,632. The production of grindstones and pulpstones, garnet, pumice, and grinding pebbles increased, and that of emery, corundum, diatomaceous earth and tripoli, and tube-mill lining decreased. American millstones are still mainly made of quartz sandstones and conglomerates, but some are made of granite; the production in 1918 was greater than it had been for about 30 years. Pulpstones are heavy grindstones used for grinding wood in the manufacture of pulp and paper; the increased production of 26 per cent. compared with that in 1917, and of 320 per cent. over that for 1915, was probably due to the difficulty and high cost of importing stones from Great Britain.

North Carolina is the sole producer of corundum; the output in 1918 was about 10,000 short tons, or 38 per cent. less than in 1917. Of diatomaceous earth, 2965 short tons was sold, but there was also a considerable production for special uses which the Geological Survey is not at liberty to report. Owing to the embargo placed upon all overseas imports of pumice, domestic supplies were developed and these proved quite satisfactory except for the finest work, viz., lithography and metal-polishing prior to plating; the production was 30,637 short tons. Similarly, the cutting off of supplies of "flint liners" for tube-mills (blocks cut from flint or other hard silicious rock) developed a domestic supply which proved quite satisfactory.

Three different kinds of artificial abrasives are manufactured in the United States:—(1) Metallic abrasives, including "diamond crushed steel" (crushed crucible steel), "angular grit" (crushed chilled iron), and "crushed cast iron" all made by the Pittsburgh Crushed Steel Co.; (2) silicon carbides, including carborundum, crystolon, and carbolon; (3) aluminium oxides, including alundum, aloxite, etc. In 1917 the production of artificial abrasives amounted to 57,911 short tons, valued at \$8,137,242; the production figures for 1918 are incomplete.

**Peat in 1919.**—The quantity of peat, excluding raw and including air-dried and mechanically-dried peat, produced in the United States in 1919 was 69,197 short tons, valued at \$705,532, an average price of \$10.20 per ton. This represents a decrease of 35 per cent. in quantity compared with the 1918 production. Most of the output was used for fertiliser purposes, either directly or as a nitrogenous ingredient of compound fertilisers; 6402 t. of carbonised peat was used as a constituent of cattle foods and a small amount as fuel.—(*U.S. Geol. Surv.*, Oct. 14, 1920.)

## SOUTH AFRICA.

**Coal By-Products.**—According to the General Manager of Railways, considerable attention is being given by the collieries to the recovery of coal by-products. There are large accumulations of waste and low-grade coal at the various collieries which, though not suitable for sale purposes, can be treated for by-products. The utilisation of this waste material has received greater attention during recent years, and it is said that the percentage of waste has been halved since 1910. Even to-day the waste coal is estimated at 1 million tons per annum. Much experimental work has been done recently on the testing of coal for the production of coke and tar and the distillation of oils and creosote. The companies are hopeful of the outcome of these by-product industries, and there is

certainly a large field for development. The building up of a trade in coal hy-products, which can be used in such a variety of ways, would be of inestimable value to the Union and would no doubt result in the creation of a number of new industries.

**Mineral Production in 1920.**—The Imperial Mineral Resources Bureau has received from the Department of Mines and Industries of South Africa the following statistics relating to the production of minerals and metals in 1920:—

	Weight. Fine oz.	Value. £
Gold (output) .. .. .	8,158,455	34,654,922
Silver (sales and shipments) .. .. .	892,205	224,769
	Carats.	
Diamonds (output) .. .. .	2,551,665	14,762,956
	Tons.	
Coal (total coal sold) .. .. .	11,473,452	4,506,572
Copper ore and concentrates (sales and shipments) .. .. .	10,874	445,007
Tin concentrates and metallic (sales and shipments) .. .. .	2,451	446,234
Other base minerals (output) .. .. .	—	153,694
Total value .. .. .		55,194,204

\* Value calculated at the standard rate of £4.24773 per fine ounce.

#### CANADA.

**Projected New Sodium Carbonate Plant.**—The Soda Mining and Products Company is to erect a new evaporating plant at Soda Lake in British Columbia. Soda Lake is situated 3700 feet above sea level, is 100 acres in extent, and its waters contain 6 per cent. of sodium carbonate.

**Mineral Production in 1920.**—The Chief of the Division of Mineral Resources and Statistics for Canada states that, viewed as a whole, the mineral industry was prosperous in 1920, although in Central Canada the fuel supply was inadequate and transport facilities insufficient; and in many camps labour was scarce and expensive. The production of copper, nickel, zinc, coal, etc., quickly recovered from the depression of 1919, but the present trade slump began too late in 1920 to have much effect on the mineral production of that year. Canadian mineral production has risen in value from \$176,686,390 in 1919 to roughly \$200,000,000, thereby closely approaching that for the maximum year, viz., \$211,301,897 in 1918.

With an output of 27,455 long tons, nickel production showed an increase of 38 per cent. over that in 1919; the annual production of this metal for some years hence will be limited only by the demands of the market, as reserves are ample and the smelting capacity is in excess even of the highest war-time production (41,295 t. in 1918). There was an increase of 10 per cent. in the production of copper (36,830 t.), but the output of lead, including lead ingots and lead recovered from ores exported decreased to 19,565 t. British Columbia produced 13,524.5 long tons of refined zinc out of an estimated total of 18,750 tons (14,373 t. in 1919). The value of the gold output is estimated at \$16,000,000 (\$15,850,423 in 1918), over two-thirds of which was produced in Ontario. There was a general reduction in the output of silver; British Columbia produced 13,500,000 oz. compared with 16,020,000 oz. in 1919. Shipments of asbestos in 1919 included 136,765 t. of fibre and 22,471 t. of powder, a production which, it is estimated, was exceeded by 25 per cent. in 1920. The estimated output of coal, 16,000,000 short tons, was about 18 per cent. greater than in 1919, but the production of iron ore did not exceed 120,000 short tons, compared with 197,170 short tons in the previous year. There were larger outputs of pig iron and steel, the production in 1919 and 1920 being:—Pig iron, 917,781 and 1,080,000 short tons; steel ingots and steel castings, 1,030,342 and 1,220,000 short tons. (*Bd. of Trade J.*, Feb. 17, 1921.)

#### AUSTRALIA.

**Mineral Production in 1920.**—The following table, showing the mineral production of Australia in 1920, has been compiled by the Imperial Mineral Resources Bureau:—

	Production. Long tons.	Exports. Long tons.
Copper—		
Bar copper .. .. .	24,069	28,612
In blister copper .. .. .	2,000	2,000
In copper ore .. .. .	115	115
Lead .. .. .	4,077	50,069
In lead bullion .. .. .	1,939	1,798
In concentrates .. .. .	4,122	4,122
Zinc .. .. .	9,665	5,689
Zinc concentrates .. .. .	—	52,732
Tin .. .. .	4,108	3,015
Pig iron .. .. .	344,000	22,657
Arsenic .. .. .	1,202	582
Silver .. .. .	701,177	341,001

The zinc concentrates contained approximately 11 oz. silver per ton, 47 per cent. zinc, and 7 per cent. lead.

**Glassware Manufacture.**—The annual imports of glassware into Australia are so great as to indicate that locally manufactured supplies fall far short of the demand. In view of the absence of any domestic glassware manufactory in Western Australia, the discovery at Wanneroo, near Perth, of a particularly high grade of glass-making sand is considered an excellent opportunity for establishing a useful secondary industry. Reports of the test of a bulk sample of the sand carried out in a well-known works in New South Wales fully confirm the opinion of the Government Mineralogist, Dr. G. S. Simpson, whose analysis gave:—Ignition loss, 0.08%; on ignited sand  $\text{Fe}_2\text{O}_3$ , 0.4%;  $\text{CaO}$  nil,  $\text{Al}_2\text{O}_3$  nil,  $\text{TiO}_2$ , 0.007%, and  $\text{SiO}_2$ , 99.81%. Colour of ignited sand, white.

The owners of the mineral claim have decided to form a company to work the deposit, and it is intended to secure the most expert advice that can be obtained. The raw material is so abundant and the quality so good that success seems assured. Western Australia is so situated that there is a big market in the Dutch East Indies and the Federated Malay States for goods of the description that it is proposed to manufacture, and there is every reason to believe that a good foreign market can be established, apart altogether from the home demand.—(*Ind. Austral.*, Dec. 9, 1920.)

#### BRITISH INDIA.

**Chemical Industries in Western India.**—The annual report of the Director of Industries of the Bombay Presidency for 1919-20 states that Dr. A. N. Meldrum and Mr. D. M. Gangoli have written the first bulletin of the Department on the preparation of sulphuric casein. A large amount of casein is produced in the Bombay Presidency, but it is very crude, and the authors are working to produce a high-grade product. The hand-made paper industry at Ahmedabad has been studied by Dr. Meldrum and Mr. Bharucha, and efforts have been made to popularise the unglazed paper for filter work and as an absorbent for drying plants. The investigation of the Kharaghoda bitters has been continued and a promising sample of Epsom salts has been obtained by Mr. A. J. Turner, who has also examined the economic production of refined salt from ordinary salt crystals. Favourable results were obtained, and a plant will, it is hoped, shortly begin working. The extraction of bromine from the bitters at Kharaghoda and Aden has been investigated, but appears economically unpromising under present circumstances. The wood-distillation industry has been closely investigated and the conclusion was reached that it was inadvisable to erect a fully-equipped plant, as the demand for by-

products had decreased. The demand for charcoal, however, is rapidly growing, and a plant producing charcoal of good quality, together with one or two by-products for which there is a market, would have a chance of success. Possible developments of hydro-electric power in the Presidency are being examined, as the price of Indian coal, like that of oil fuel, is increasing and supplies and labour are difficult to obtain. Power alcohol has also been considered, and a plant for producing a mixture of alcohol and ether is being completed in Hyderabad State, but development is hindered by the circumstance that any scheme to distil alcohol on a large scale in India would have to reckon with the competition of cheap alcohol imported from Java.—(*Indian Tr. J.*, Jan. 21, 1921.)

#### GENERAL.

**Sugar Beet in England.**—It now appears certain that the beet-sugar factory of Home-Grown Sugar, Ltd., the company in which the Ministry of Agriculture holds half the share capital, will be ready by next autumn; much of the plant ordered from France has already arrived. Supplies of sugar beet for the present year have been contracted for, and although the factory will be able to deal with 600 tons of beet per day for 100 days, the tonnage for the first year is to be limited to 20,000, grown on 2365 acres, in order not to overload the factory, while the machinery is new, and to allow the staff to be adequately trained by the French specialists engaged. There are 425 farmers in Nottinghamshire and Lincolnshire who are cultivating an average of 5½ acres each, and it would have been possible to have doubled the acreage actually contracted for. For the reasons stated above, only 200 of the 400 acres originally contemplated are being grown on the Kilham Estate. The price payable per ton of beet delivered at the factory has been fixed at £4, equivalent to £3 17s. 6d. delivered on rail, and as experimental trials showed that the cost per acre of 12 tons (with an average sugar content of 20 per cent.) delivered on rail was £29 3s. 4d., there should be sufficient margin to induce the farmers not to limit expenditure on fertilisers, cultivation, and supervision, all factors that materially affect sugar content as well as yield.—(*Official.*)

**Asbestos, 1913—1919** (H.M. Stationery Office, 34 pp., 1s.) is one of the most interesting pamphlets yet issued by the Imperial Mineral Resources Bureau. The name "asbestos" is applied to several fibrous silicate minerals differing from one another in chemical composition and physical properties. The principal asbestos mineral used in commerce is *chrysotile*, or serpentine asbestos, which is essentially a hydrated silicate of magnesium. It has great tensile strength and flexibility, is unattacked by acids, and is remarkably fire-resisting, although at 500° C. it becomes dehydrated and loses its tensile strength. It is a good insulator for both heat and electricity and is well adapted for spinning. *Crocidolite*, or blue asbestos, is a silicate of iron and sodium and is found in large quantities only in South Africa. It has great heat-insulating capacity, but its fire-resisting properties are poorer than those of *chrysotile* owing to its comparatively low fusibility. Asbestos cloth, rope, felt, etc., can be manufactured from *crocidolite* fibre, as also can sheets, tiles, cement, and other building material.

The commercial value of asbestos depends chiefly on tensile strength, flexibility, length and fineness of fibre, dehydrating temperature, and temperature of fusion, but other factors of importance are heat-insulating capacity, electrical insulating power, and colour. For spinning and weaving into fabrics, ropes, and engine packing, the qualities of first importance are tensile strength, fire resistance, and length of fibre. Although of greater monetary value, such articles form only a small proportion of

the total output of asbestos manufactures. The larger proportion of the asbestos mined is used for sheets, tiles, slabs, millboard, joint-packing, etc. in the building, engineering, and electrical industries. Finely ground *anthophyllite* (silicate of magnesium and iron), low-grade *tremolite* (silicate of magnesium and calcium), and the residues from the *chrysotile* mills are used extensively in the manufacture of asbestos cements, fireproof bricks, tiles and other roofing material, paints, etc. Asbestos wool mixed with a solution of sodium silicate makes a fireproof cement of great strength.

The largest and most valuable deposits of asbestos occur within the British Empire. Canada has long been the chief producer of *chrysotile* asbestos (137,000 tons in 1919, or about 80 per cent. of the world's output). Rhodesia is second with about 8700 tons, but, arising out of the keen demand for Rhodesian fibre, a policy of rapid and extensive development is being pursued in that country. The Union of South Africa, which is the only producer of *crocidolite*, exported 3500 tons in 1919. Russia was formerly an important producer (17,200 in 1913), and other producing countries, in order of importance, are Australia, Cyprus, United States, India, and Italy. The price of Canadian No. 1 is stated to have risen from \$300—\$350 in 1913 to \$2500—\$3000 per ton in 1919. Cheaper grades range down to \$25—\$30 for "C short fibre." Most of the Rhodesian and South African asbestos is exported to Great Britain, but the Canadian output is exported largely to the United States, only relatively small quantities being sent to the United Kingdom.

**The International Chemical Union.**—The full report of the meeting which was held in Rome in June last has now been published from the offices of the secretariat at 46, Rue des Mathurins, Paris. (*Cf. J.*, 1920, July 31, 251 n.)

**Importation of Dyes and Dye-Intermediates.**—The list of products covered by the Dyestuffs (Import Regulation) Act, which has been drawn up in consultation with the Dyestuffs Advisory Licensing Committee, is now available and may be obtained on application either to the Board of Trade, Industries and Manufactures Department, Great George Street, Westminster, London, S.W. 1, or to the Dyestuffs Advisory Licensing Committee, Board of Trade, Danlee Buildings, 63, Spring Gardens, Manchester.

**Chemical Industry in Belgium.**—The shortage of fuel and raw materials has prevented the Belgian chemical industry from recovering its pre-war position, but exports have been increasing. Supplies of sulphuric acid are very short, the production in the Gand district being at the rate of 40,000 tons per annum, compared with the pre-war output of 125,000 t., and owing to the strike the Vieille Montagne works are producing at only 40 per cent. of their capacity. The oil industry is in a difficult position on account of the high prices and shortage of raw materials, and the rubber industry is similarly situated. Full time, however, is being worked in the artificial silk industry, and export orders have been plentiful.—(*L'Ind. Chim.*, Dec., 1920.)

**The Belgian Iron and Steel Industry.**—In spite of the active reconstruction work carried on since the armistice, the Belgian iron and steel industry is still far from attaining its pre-war position, mainly because a large proportion of the labour has had to be diverted from production to repair work. Out of 51 blast furnaces, 21 are producing at the rate of 65 per cent. of the total 1913 production and 7 are ready for blowing in. There is sufficient coke for present needs, but gas coal for generators and Martin converters is in short supply. By the autumn of 1920 the iron and steel industry was producing 51 per cent. of its pre-war output of pig-

iron, 69 per cent. of finished iron, 54 per cent. of steel ingots, and 62 per cent. of finished steel. Since May, 1920, the high prices have led to a reduced demand, and French, Belgian, and Luxemburg producers have had to agree to fix the price of pig iron at 500 fr. per ton. Further, the re-adjustment of labour costs to falling prices is a difficult problem, and, in spite of the progress made in reconstruction, the present market outlook provides no incentive for further expansion (*cf. J.*, 1921, 15 R).—(*U.S. Com. Rep.*, Feb. 7, 1921.)

**The Belgian Glass Industry.**—The Belgian glass industry, and in particular the plate-glass industry, profited by exceptionally favourable conditions during the early months of 1920, but now there is a general lack of new business and plate-glass manufacturers are curtailing production in order to maintain prices; the production of plate glass in the first eight months of 1920 was 98.5 per cent. of that in the same period of 1919. Window-glass manufacture is also seriously affected by the decline in demand, and prices have dropped by nearly 50 per cent. The problem of diminishing production costs by reducing labour charges has become so acute that arrangements are being made to introduce a process for drawing glass mechanically. The flint-glass industry has been less affected by the prevailing depression. There are at present 35 plants producing table-ware, and some of them, especially the Cristalleries Val St. Lambert, manufacture the highest and most artistic grades of cut-glass. Cut-glass and table-ware are exported to the United States to the value of about 500,000 fr. monthly. Much of the effectiveness of Belgian flint glass depends on the skill of the workmen, machinery being but little employed. The wages of glass workers and cutters vary from 25–30 fr. and 35–40 fr. a day, according to the district; increases have been at the rate of 320 and 400 per cent., compared with an increase of 370 per cent. in the cost of living since 1914. The exports of glass (in metric tons) during the first 8 months of 1920 were: Rough plate, 662; polished plate, 22,463; window glass, 120,466; common glass, 67; table-ware and cut glass, 15,225; bottles, demijohns, etc., 10,810. There has been an enormous increase in exports to the United States during the past year, viz., 3599 t. in the first ten months, compared with 162 t. in the same period of 1919.—(*U.S. Com. Rep.*, Jan. 21, 1921.)

**The Window-Glass Industry in Sweden.**—Competition from Belgium has very seriously affected the Swedish window-glass industry; only three or four factories are now working. It is reported that Belgian firms are dumping window-glass on the Swedish market at prices below the cost of production, and that the industry has applied to the Government for higher tariffs and temporary import prohibition. During the period April to July, 1920, 1870 metric tons of window-glass was imported, and the home production was 2130 t.—(*U.S. Com. Rep.*, Feb. 3, 1921.)

**The German Fluorspar Industry.**—The largest fluorspar deposits in Germany are situated in the Hartz district, Upper Palatinate, the Thuringian Forest, and the Black Forest; that found in the Upper Palatinate contains 95–98 per cent. of calcium fluoride and relatively little silica. The quantities supplied to the various consuming industries before the war were distributed as follows:—Iron and metal smelting, 79–84%; glass industry, 10–15%; chemical, enamel, and optical industries, 5% each. The use of fluorspar in the iron industry has increased since the armistice, as it effects an economy in fuel. Owing to the depreciated currency, German producers are able to sell fluorspar for exportation at lower prices than any of their foreign competitors.—(*U.S. Com. Rep.*, Feb. 9, 1921.)

**Carbon Dioxide as Fertiliser.**—The possibility of manuring plants with furnace gases containing carbon dioxide by Dr. Riedel's method appears to be established, as his results have been confirmed by independent researches and trials by Bornemann and Reinau. Dr. Claassen, however, criticises the practicability of the method and its economy when used on a large scale. The plant for distributing the gases is inexpensive; all that is required is the main pipe and smaller pipes for conducting the gases to the different parts of the fields. Then by opening or closing the valves, the farmer can treat his plants with larger or smaller amounts of carbon dioxide according to the weather and the state of the plants. From the results it appears that lupins after treatment with gas gave, in the dry state, a yield 2.9 times greater than an untreated crop. A sugar-beet crop was increased by 85 per cent., with 1½ per cent. more sugar.—(*Chem.-Z.*, Feb. 15, 1921.)

**The Potash Industry in Germany.**—A resolution to increase the inland prices of potash compounds by 50–55 per cent. was considered by the Potash Council (Reichskalirat) on February 1, but not carried as the voting for and against was equal. The representatives of agriculture and labour voted against the resolution. The case for increasing the prices included the following considerations. In spite of enormously increased costs of production, potash prices have not been raised since December, 1919. Whereas the price of potash has only risen five to sixfold since 1913, that of nitrogen has risen 9 times, phosphoric acid 19–20 times, and agricultural produce 104 times. Costs in the potash industry have increased since 1913 as follows:—Lignite 20 times; coal 13 times; wages 10 times; and costs of materials have risen far more than any of these items. The doubling of the railway freights in March, 1920, hit the industry particularly hard because coal from the Ruhr district being unavailable (under the Spa Agreement), the potash works have to use three times the amount of lignite, and this has to be transported distances up to 250 kilom. About 88 per cent. of the losses on inland sales in 1920 were made good by the export trade, but this was no longer possible in 1921. Employees have been dismissed from 21 of the works, further dismissals are taking place daily, and working hours have been curtailed at 46 works. The Labour Minister has just notified further increases in wages and fuel which, it is estimated, will entail additional charges on the industry of 42 million and 17 million marks, respectively. The Potash Syndicate is anticipating the closing down of many more works.—(*Z. anorg. Chem.*, Feb. 2, 1921.)

**The Balsam-Resin Industry in Germany.**—A translation of an article on the German balsam-resin (Balsamharz) industry by Vice-Admiral D. F. Funke (*Handelszeitung*, Feb. 10, 1921) has been made available; together with supplementary information, by the Department of Overseas Trade.

Prior to the war the production of resin was increasing and imports were decreasing. Thus 113,481 tons, of which the United States supplied 78 and France 19 per cent., was imported in 1912, and 96,265 t. (U.S.A. 80, and France 16.5 per cent.) in 1913. The exports were 17,060 t. in 1912, and 25,893 t. in 1913, Austria-Hungary taking 31.4 and 30.5 per cent., and Russia 29.6 and 26.5 per cent., respectively. During the war the industry, controlled by the German Government and under the supervision of technical experts, was developed to a remarkable extent. Government control was abolished at the end of 1919, and the new companies that had been formed to treat the raw material were placed in a very precarious position. However, as a result of the intervention of the German Minister of Economics, an agreement was reached



between the refiners and the consumers, under which a new company, the Deutsche Harz-Gesellschaft, was formed to take over the distribution of all the resin produced. All risks were borne by the consumers, who agreed to order what they required annually at a price to be based on the world's market prices at the time of the order. Resin manufacturers and merchants, however, have met with opposition from the Forest Authorities, who apparently have done all they could to obstruct the industry. Various excuses were made to diminish production, private forests were closed down, and finally a charge of 8 marks per kg. was made for the balsam-resin. Owing to the heavy fall in price in 1920, the demand ceased and representations were again made to the Minister of Economics which resulted in the price being reduced to 6 marks per kg. This did not satisfy the consumers, and efforts were then made to obtain statistics of the cost of production to the forest authorities. No figures were available for 1920, but official figures for 1919 showed that the Prussian forest authorities in 1913 made a profit by selling at 3 marks per kg. Further, the cost of production was so low that with a production of 1307 kg. per hectare (10.4 owt. per acre) a profit of 2.57 marks per kg. was made. During the war the forests in the Oberost district actually produced 1500–2000 kg. per hectare, and these figures are now being put forward to prove that the forest authorities are over-charging.

**The Beet-Sugar Industry in 1920.**—In an article on the progress achieved in various branches of the beet-sugar industry during 1920 (*Chem.-Z.*, Feb. 22, 1921), Prof. E. O. von Lippmann insists that this industry should be thoroughly re-organised. The American statement that single-germ seeds show increased resistance to animal and vegetable pests is not corroborated; opinions, in fact, differ as to the desirability of growing these single-germ seeds at all. Concentrating syrups on an extensive scale by means of low temperatures is, according to Pokorny, Linsbauer and others, impracticable both from the technical and economic standpoints. Stenzel's process for fermenting waste waters by means of yeast, with the object of manufacturing food-material, does not offer any prospect of success. Opinion is unanimous in Germany, France, Bohemia and other countries that the usual chemical control in beet-sugar factories is inadequate. Teyssier calls attention to the inaccuracy in calculating the "rendement," or net sugar, of samples of raw sugar from ash determinations. British, American and most French sugar chemists have declared themselves in favour of adopting 20 grams as the new normal weight. Zeller, in his exhaustive treatise on the relation between cane and beet sugar, comes to the conclusion that the world's sugar requirements cannot be satisfied by cane sugar alone since its supply is limited by technical, industrial and economic considerations. According to Bartens, the world shortage on September 1, 1920, amounted to 6 million tons, and in his opinion only the re-establishment of the beet-sugar industry can relieve the situation. The conditions in Czecho-Slovakia are the most promising. In Russia and Russian Poland all progress is checked for many years to come. In France also it will take years before the efforts now being made to re-organise the industry can materialise. In Germany, owing to all the reserve stocks having been exhausted by September 1, 1920, the price of refined sugar ultimately rose to 40 marks per kg. Moreover, according to a U.S. Consular Report of February 4, 1921, the total sugar production for the year 1919–20 was only 733,000 t., as compared with 1,346,000 t. in the previous year. To meet her domestic requirements in 1919–20, Germany had actually to import 57,000 t., or more than twice as

much as during the preceding year, and a larger importation was only avoided by the existence of reserves from the previous season amounting to 216,000 t. The average yield of 7.5 t. per acre was the lowest for many years. Only 269 beet-sugar factories are now working in Germany.

**Emil Fischer Bequest.**—The late Prof. Emil Fischer left the sum of 750,000 mk. to the Prussian Academy of Sciences for the establishment of an "Emil Fischer Foundation"; the income from the fund is to be applied to the maintenance of young German chemists studying organic, inorganic, or physical chemistry.—(*Chem.-Z.*, Jan. 22, 1921.)

**Technical Education in Germany.**—The number of students attending the Technical "Hochschulen" in Germany, including 1200 at the Köthen Polytechnic, is 21,000, compared with 18,900 in the winter semester of 1920–21, and 12,200 before the war. In electrotechnology the number of students has increased from 1300 to 3229; in mechanical engineering from 3100 to 6540; in chemistry, natural science, and pharmacy from 1544 to 3174; and in mining and metallurgy from 576 to 785. There are 3209 students at Charlottenburg, 2923 at Munich, 2591 at Hanover, 2264 at Dresden, 2206 at Darmstadt, 1631 at Stuttgart, 1491 at Karlsruhe, 1088 at Aachen, 872 at Brunswick, 837 at Breslau, and 779 at Dantzig.—(*Chem.-Z.*, Jan. 22, 1921.)

**New Carbide Factory in Hungary.**—The first carbide factory to be established in Hungary has been erected at Felsőgalla by the Ungarische Allgemeine Kohlenbergbau A.-G. and is now ready to start; it will have an initial output capacity of 80 wagon loads per annum, and will utilise charcoal as coke is in short supply. The chief consumers of carbide in Hungary include the State railways, mining enterprises, the oxygen industry, etc., and it is hoped that a large part of the country's needs in carbide will now be covered.—(*Handelsmuseum*, Feb. 10, 1921.)

**Sicilian Phosphate Deposit.**—A deposit of phosphate, extending over 10 kilometres, occurs on the shore at Donna Lucata, south of Scicli (Syracuse). The phosphates, present in the form of nodules of varying size, contain an average of 33 per cent. tricalcium phosphate (hand-picked specimens), and the reserve of utilisable mineral is stated to amount to 2 million metric tons.—(*G. Chim. Ind. ed. App.*, Feb., 1920.)

**Graphite Production in Madagascar.**—The French Colonial Ministry reports that trade conditions were so unfavourable during 1920 that no new graphite deposits were opened up, that operations at some of the old deposits were abandoned, and that the mineral has recently been sold at a loss. The export of graphite has declined from 27,833 metric tons in 1917, to 15,015 t. in 1918, and 4056 t. in 1919.—(*U.S. Com. Rep.*, Feb. 5, 1921.)

**Tung Oil in China.**—Tung oil, or Chinese wood oil, is obtained from the nut of certain species of *Aleurites* which grow luxuriantly in hilly districts and seem to thrive in poor soil. The oil is usually extracted in the interior and shipped to Hankow, where it is stored. Its high price often leads to adulteration, soya bean, groundnut, sesame and cottonseed oils being used for this purpose. The exports from China from 1913 to 1919 were as follows:—1913, 37,598 tons; 1914, 26,164 t.; 1915, 18,473 t.; 1916, 30,665 t.; 1917, 23,890 t.; 1918, 29,098 t.; 1919, 36,515 t. There is room for considerable improvement in the methods of cultivation, harvesting and expressing, and the installation of modern machinery would, it is stated, increase the production by at least 10 per cent. Attempts to cultivate the tung tree in the United States are said to have been successful.—(*U.S. Com. Rep.*, Feb. 9, 1921.)



## CORRESPONDENCE.

## A BRITISH CHEMICAL NEWS SERVICE.

SIR,—There is one notable omission from the literary activities proposed by the Federal Council for Pure and Applied Chemistry, recently made public. Sound suggestions are put forward for making scientific information available to chemists, but nothing is said as to one most important aspect of chemical publicity, namely, the means for keeping the lay public fully informed of the activities of chemists and their bearing on public welfare, for the success of both pure and applied chemistry is ultimately dependent upon an active and well-informed public opinion on all that relates thereto.

You, Sir, have already put forward the suggestion some time ago (*cf.* J., 1918, 27 B, 85 B) that the question of publicity should be taken up by technological and scientific societies on this side of the Atlantic, and I feel that the time is now more than overdue for a revision of our present policy of "laissez faire" in this matter. It is well known to chemists how strong a position the American Chemical Society now holds in forming public opinion in the United States, and there are more than sufficient indications in this country that we also need to make use of the medium of publicity for urging necessary reforms in order to ensure that an adequate weight of educated public opinion will be felt whenever chemical matters come up for consideration, whether the objective be peace or war.

The activity of the American Chemical Society in this direction forms so strong a contrast to our neglect that perhaps I may be permitted to quote from a recent issue of the *Journal* of the American Chemical Society:—

"The A.C.S. News Service continues to grow in usefulness. More and more it is finding a definite and accepted place in the newspaper offices of the country and, under the increased appropriation recently granted, it is proposed to extend this work as far as possible into the field of the weekly newspapers, thereby increasing more and more the public understanding of the significance of chemistry to the nation. Much time has been required to get the monthly clip sheet issued by the News Service thoroughly established. The first attempts to include in this clip sheet textually-reproduced sections of the *Journal* did not yield very great success. This was then modified in form to cover popular treatment of articles appearing each month in the *Journal*, and the clippings being received show that this feature is now on a thorough basis and is being widely made use of by the press."

[The clip sheet referred to and the weekly bulletins are being sent to 900 of the leading American newspapers.]

To come to definite suggestions, the Chemical Industry Club might form a nucleus for the project, and weekly meetings might be arranged at the Club, at which a number of typed or printed slips could be distributed to the representatives of various papers and news agencies, with a word of explanation as to the possibilities of each article; the journalists could be trusted to make the best use of such free copy once they understood the nature of the work. By degrees the Club might be made a centre for all inquiries of a chemical nature, and with the formation of a definite *liaison* between the press and chemistry the news service would soon attain to very respectable dimensions. Now is the time to make the necessary preparations, and I would suggest that a meeting be called of all those interested in order to discuss ways and means for getting such a British Chemical News Service under way.—I am, Sir, etc.,

FREDK. A. MASON.

Manchester, March 14, 1921.

## PARLIAMENTARY NEWS.

## HOUSE OF COMMONS.

*Trade in Dyes and Dyestuffs.*

In a written answer to Major Barnes, Sir P. Lloyd-Greame stated that the imports into and the exports from the United Kingdom of coal-tar dyes and dyestuffs during 1920 were as follows:—

	IMPORTS. Tons.	EXPORTS.	
		U.K. manu- factures. Tons.	Foreign and colonial manu- factures. Tons.
Coal-tar intermediates, including aniline oil and salt, and phenylglycine .. ..	2,487.5	3,668.4	201.75
Coal-tar dyes:—			
Alizarin .. ..	600.4	372.2	1.05
Indigo, synthetic .. ..	715.55	690.9	158
Other dyes .. ..	8,516.95	5,048.65	195.25

The imports were valued at £8,365,294, the exports of United Kingdom manufactures at £3,258,133, and exports of foreign and colonial manufactures at £305,555.—(Mar. 8.)

*Coal Mines (Decontrol) Bill.*

This Bill was read a third time on March 18. Its object is to put an end to financial control over the coal-mining industry on March 31, 1921, and to repeal as from April 1, 1921, Section 3 of the Mining Industry Act, 1920, which gives the State power to control prices, export, and wages. Further, it is proposed to divide the period of operation of the Coal Mines (Emergency) Act, 1920, into two periods, in the first of which the owners' profits are to be limited to the pre-war standards, and in the second the owners are to pay the surplus above or receive the deficit below nine-tenths of their pre-war standards, instead of the whole.

*Opium.*

Mr. Montagu informed Mr. Gilbert that the agreement for the supply by the Government of India to the Government of Hongkong of 10 chests of opium per month at a fixed price had been renewed for a further period of five years as from January 1, 1921.—(Mar. 9.)

*Dangerous Drugs.*

Mr. Shortt, in reply to Mr. Woolcock, said that he had decided to appoint a small committee to consider and report what further modifications could safely be made in the draft Regulations, having regard to the principle that the use of the drugs under the Dangerous Drugs Act must be safeguarded in the public interest.—(Mar. 9.)

*Coal Mining Industry.*

Mr. Bridgeman gave the following statistics relating to coal mining in a written answer to Mr. G. Lambert:—

	1913. s. d.	1920 s. d.
Cost of raising an average ton of coal:—		
Wages .. ..	8 4	29 5½
Timber and stores .. ..	1 10	5 4½
Other costs .. ..	0 5½	2 3½
Royalties .. ..		0 7½
Total cost .. ..	8 7½	34 1½
Number of persons engaged .. ..	1,110,884	1,204,300
Amount of coal raised .. tons	287,412,000	229,295,000
Amount of coal raised per person engaged .. tons	259	190
Coal exported as cargoes .. tons	73,400,118	24,931,853
Coal exported as bunkers .. tons	21,031,550	13,840,360

In 1913 the owners' profits were at the rate of 1s. 6d. per ton, and the standard profits payable in 1920 are estimated to be at the rate of 2s. 6½d. per ton.—(Mar. 11.)

**Basic Slag Supplies.**

Sir R. Sanders informed Mr. A. T. Davies that there was no shortage of basic slag, as the decrease in production within the last two months had coincided with a still greater reduction of demand, and the stocks which had accumulated were twice as large as those held on the same date a year ago. Supplies of phosphatic fertilisers, including superphosphate, basic slag, and ground mineral phosphate for the coming season would probably equal, if not exceed, the supplies in any previous year.—(Mar. 17.)

**Anglo-Persian Oil Co.**

Mr. Chamberlain informed Col. Wedgwood that the Government holds two-thirds of the ordinary shares and 1000 out of a total of 8,500,000 preference shares in the Anglo-Persian Oil Co.; there was no intention of allowing the voting control over the company to lapse.—(Mar. 14.)

**German Reparation (Recovery) Bill.**

The German Reparation (Recovery) Bill, which was read a third time on March 18, provides that 50 per cent. of the money due as payment for German goods, or such percentage as the Treasury may prescribe, shall be paid, not to Germany, but to the Treasury through the Customs on account of German reparation. German goods are defined as (a) goods first consigned from Germany, and (b) goods consigned from elsewhere of which less than 25 per cent. of the value is attributable to production outside Germany. Transhipment goods are not affected. The Bill also contains provisions relating to valuation of goods, contracts, etc.

The Prime Minister, in reply to Mr. Pennefather, stated that the Bill would not in any way affect the promised legislation relating to dumping, collapsed exchanges, and key industries.—(Mar. 15.)

**OFFICIAL TRADE INTELLIGENCE.**

(From the Board of Trade Journal for March 10 and 17.)

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W. 1, from firms, agents, or individuals who desire to represent U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number:—

Locality of Firm or Agent.	Materials.	Reference Number.
Canada .. ..	Tiles .. ..	301
" .. ..	Pure iron sheets for the manufacture of tanks and roofing .. ..	329
" .. ..	Whitening .. ..	335
South Africa .. ..	Earthenware, china, mirrors .. ..	304
Belgium .. ..	Metals, rubber .. ..	309
" .. ..	Lubricating oils .. ..	310
Bulgaria .. ..	Hides, textiles .. ..	342
Italy .. ..	Oils, fats .. ..	349
" .. ..	Crude leather .. ..	350
" .. ..	Machinery for rubber manufacture .. ..	351
Latvia .. ..	Raw materials for soap making, soda, potash, tallow, coconut oil .. ..	313
Poland .. ..	Chemicals, raw materials for tanneries, leather .. ..	353
Spain .. ..	Chemicals, colours, paint, varnish .. ..	319
Argentina .. ..	Cheap glassware .. ..	358
Panama Republic .. ..	Vegetable oils, paper .. ..	361

**TARIFF, CUSTOMS, EXCISE.**

**Argentina.**—The export duty on certain hides is suspended until February 2, 1922.

**Australia.**—Glycerin, crude and refined, for the manufacture of explosives may be imported duty free under the British Preferential Tariff.

**Austria.**—As from March 1 customs duties when paid in bank notes must be paid at 70 times the rates prescribed by the tariff.

**Belgium.**—Among the articles under the control of the Ministry of Industry, Labour and Supply that still require export licences are alcohol, matches, butter, lard, fats, malt, yeast, sugar, syrups, and molasses.

Authorisation of this Ministry is still required for the import of butter, malt, sugar, syrup, and molasses.

**British India.**—The general *ad valorem* duty of 7½ per cent. has been increased to 11 per cent. as from March 1. The customs duty on matches is fixed at 12 annas per gross of boxes, and that on imported liquors has been increased by 3 annas per degree of proof per gallon. The *ad valorem* duty on foreign sugar has been raised to 15 per cent.

**Bulgaria.**—The surcharge on customs duties when paid in paper currency is now 900 per cent.

**Chile.**—The increased customs duties take effect as from February 23. Condensed milk, cocoa, sugar, petrol, and oils are exempted from increase.

**French Equatorial Africa.**—A copy of the Decree establishing new customs regulations may be seen at the Department, 18, Queen Anne's Gate, London, S.W. 1.

**Grenada.**—The complete text of the revised schedules of Import and Export Duties may be seen at the Department.

**Italy.**—The stamp tax on perfumery, medicinal specialities, alcoholic beverages, and mineral waters has been doubled.

**Kedah.**—Export duties have been modified on, *inter alia*, rubber, rambong, damar, wood oil, tin ore, tin, wolfram, hides, bones, bricks, and tiles.

**Kenya.**—The import of condensed skimmed milk containing less than 9 per cent. of milk fat is prohibited as from February 5.

**Netherlands.**—The prohibition of the export of toluene has been removed.

**Peru.**—The export duty on vanadium ores and concentrates is fixed at £p.4.529 per metric ton, irrespective of their vanadic acid content.

A copy of the new customs tariff and customs regulations, effective as from October 15, 1920, may be seen at the Department.

**Rumania.**—Export duties have been reduced on rape seed, common soap, and toilet soap.

**St. Christopher Nevis.**—The revised schedules of customs duties came into operation on January 2. The complete text may be seen at the Department.

**San Salvador.**—The export duties on sugar, hides, and minerals have been revised.

**Senegal.**—Groundnuts may now be exported to the U.K. without restriction.

**Sierra Leone.**—The *ad valorem* import duties have been increased to 20 per cent.

The export duty on palm kernels prescribed by the Order in Council of March 29, 1920, has been revoked.

**South Africa.**—Asphalt, bitumen, pitch, and preparations thereof imported under item 76a of the customs tariff must contain less than 60 per cent. of mineral matter, and heavy oils imported under the same item must not lose more than 10 per cent. of their weight on being heated at 160° C. for 6 hours.

**Trinidad.**—The export of sugar is prohibited except under licence as from February 14.

**Tunis.**—The export tax on superphosphates is fixed at 1 franc per metric ton.

## LEGAL INTELLIGENCE.

**RAIL CONVEYANCE CLASSIFICATION OF A WASHING POWDER.**—*Joseph Crosfield and Sons, Ltd., v. London and North Western Railway Co.*

On March 4 the Railway and Canal Commissioners dismissed an application by Messrs. J. Crosfield and Sons, Ltd., that their preparation "Carbosil" should be classified in Class 2 for the purpose of railway conveyance. Until July 1, 1917, it had been so classed, but then the railway company decided to classify it as soap powder and charge for it in Class 1.

The case for the railway company was that the applicants had for years enjoyed the advantage of having "Carbosil" carried at Class 2 rates, and that their competitors had objected as all other washing powders were carried in Class 1. Applicants had refused to pay Class 1 rates because they maintained that "Carbosil" was an alkali. The amount of extra rate it was sought to obtain was about £1000 a year, and the business was growing.

In the course of the hearing, counsel for applicants admitted that in 1907-8 "Carbosil" had been advertised as a soap powder, but submitted that throughout the whole period the railway company had carried it in Class 2 with full knowledge of its composition; and it devolved upon the railway company to prove that the increased charge was a reasonable one.

Mr. Justice Lush said that the fact that "Carbosil" had been advertised as a washing powder showed that its classification in Class 1 was correct. On the point of the reasonableness of the increased charge, it was conceded that the railway company had the right to alter a classification after finding out that it had been incorrect; without deciding that question he thought that this was what had been done. He had much doubt if the charge under the Act had been increased at all, but assuming that the onus was on the railway company to prove that the increase was a reasonable one, he thought that it had abundantly proved its case. The applicants were liable for payment of any sums due.

**BREACH OF CONTRACT FOR THE SALE OF CALCIUM CARBIDE.** *Acetylene Corporation of Great Britain, Ltd. v. Canada Carbide Co., Ltd.*

The action brought by the Acetylene Corporation of Great Britain against the Canada Carbide Co. of Montreal for alleged breach of contract in respect of the supply of calcium carbide during the war was terminated on March 9 in the King's Bench Division.

Plaintiff's case was that at the outbreak of war the defendant company had agreed to supply it with calcium carbide on condition that the Corporation had sole control of the sale of all the company's products in this country. The first contract was made for the delivery of 3600 tons beginning in December, 1915, all of which was delivered, but under the second contract only 200 tons was delivered out of a total of 7000 tons. The terms of both contracts stated c.i.f. United Kingdom, and provided that if freight cost more or less than 75s. per ton, the difference should be paid by or allowed to the defendant company. After the price of carbide had begun to rise in the middle of 1916, the company commenced secretly to sell direct to the Admiralty, f.o.b. Montreal, for shipment to this country, alleging that shipping space was not procurable for sending the goods to the Corporation. The latter claimed that shipping space was available, and that it had been declined because the company was supplying to the Admiralty. Hence the Corporation had to buy carbide at £25 per ton in order to fulfil its contracts to sell to the Admiralty

at £18 per ton. In 1917 the Admiralty cancelled its contracts with the company, whereupon the latter offered to sell to the Corporation at \$20 above the contract price, as it was obtaining \$65-95 per ton from the Imperial Munitions Board in Canada. By 1919 the company had repudiated all liabilities under the contracts and had initiated a campaign for the sale of its goods in this country. The result was that the Corporation had to pay £25, £35, and £45 per ton for carbide it had contracted to supply, and that the company made large profits on goods it had contracted to sell to the Corporation at \$45 a ton. The Corporation alleged a breach in 1917 and a continuing breach afterwards.

For the defence it was submitted that owing to manufacturing difficulties and lack of shipping space, the suspensory clause contained in the contracts came into operation from 1917 onwards; and that it was not established that the carbide sold to the Canadian War Department was sent to this country and thus came under the contract.

Mr. Justice Rowlatt gave judgment for the Corporation for damages to be ascertained.

## TRADE NOTES.

### BRITISH.

**Canadian Imports.**—Despite the adverse rate of exchange that prevailed during the year, the value of imports into Canada from the United States increased very greatly, the increase in the value of imported chemicals and allied products alone amounting to about \$6,000,000 over that for the previous year. The fact that imports from the United Kingdom also increased greatly during 1920 show the splendid possibilities that exist in Canada for the development of a market for British goods. The following figures may be of interest in this connexion:—Imports from the United Kingdom into Canada for the twelve months ending November (December totals are not yet available) were valued at \$229,925,844, as compared with \$84,638,900 for the corresponding period in 1919-20; imports from the United States during the same periods were \$927,564,711 and \$725,968,553 respectively. Imports of chemicals and allied products, including such items as dyestuffs and explosives, from the United Kingdom for the period in 1920 were valued at \$27,030,165, compared with \$21,126,643 from all sources in the previous year. Exports to the United Kingdom for the period decreased, while exports to the United States increased, the figures being: to the United Kingdom, \$352,083,388, against \$514,012,960; to the United States, \$545,283,365, against \$443,416,125.

**Ashanti in 1919.**—The chief industries of Ashanti are the production of cocoa, kola, and gold. The cacao plantations suffered from drought and insect pests, factors which are limiting the extension of the crop; the production in 1919 amounted to 31,944 tons, but though this represented a decrease as compared with previous years, the quality showed a marked improvement. The kola crop was up to the average and 6171 tons was exported. Ground-nuts have not been cultivated except on an experimental scale, but large areas are available for this purpose. The total yield of dry rubber was 6150 lb.; and 108 tons of skins was exported. Gold production was maintained during the year, the yield being 99,269.76 oz., valued at £421,698, as against 99,278.85 oz., worth £421,736, in 1918. Local industries, e.g., weaving, basket-making, and pottery manufacture, are general throughout the country, but are not exported.—(*Col. Rep.-Ann.*, No. 1058, Jan., 1921.)

## FOREIGN.

**Deliveries of German Potash to America.**—The *Deutsche Bergwerks-Zeitung* of February 19 publishes a report that the Potash Syndicate intends meeting America half way in regard to the sale of potash. The unsold stocks of chloride of potassium in America amount to 20,000 tons, and a further 29,000 tons, valued at 47,421,400 mk., is lying at German harbours ready for despatch. In order to put an end to the dispute concerning the "Baisse clause," the German Potash Syndicate intends to deliver 40 per cent. of the amount of potash already delivered at a rate far below that agreed upon for the old contracts. Owing to the depreciation of the American exchange, large sums of money have been lost on the 20,000 tons lying in America, the value of which is estimated at \$1,043,357, and the German Potash Syndicate, by these supplementary deliveries, wishes to make good to a certain extent these American losses in order to create a new basis for trade.—(Official.)

**The Chinese Soap Trade.**—The use of washing soap in China is practically limited to the areas at or near treaty ports and large trading centres. In the interior a kind of vegetable soap, known as soap bean, is used in laundry work. Imported soaps were formerly widely sold in the treaty ports and outlying districts, but have now to face considerable competition from native products. Some 60,000 cases of soap are produced yearly in Hankow by four native soap works, which are equipped with little or no machinery. Foreign firms that sell soap in China require cash in advance, whereas the native manufacturer sells on consignment and allows long credits and other facilities, with the result that several foreign soaps no longer command a market. The imports of washing soap in 1913, 1915, 1917, and 1919 amounted to 1029, 916, 1099, and 468 tons, respectively. There are almost unlimited possibilities for the development of this industry, and the abundance and cheap supplies of both raw materials and labour would suggest the establishment of foreign-owned factories in China. Apparently the possibilities of developing the toilet-soap trade are even greater, and the establishment of a factory in China for this purpose would doubtless meet with success. The value of the imports of toilet soap in 1919 was £48,148.—(U.S. Com. Rep., Dec. 20, 1920.)

**Foreign Company News.—Czecho-Slovakia.**—A group of German textile manufacturers has formed a new company in Prague to manufacture artificial silk.—(U.S. Com. Rep., Dec. 13, 1920.)

**France.**—The capital of the Société Chimiques des Usines du Rhône is to be increased to 21 million francs.

The Compagnie Nationale de l'Azote (capital 12,500,000 fr.) has been formed to take over the contract made between the French Ministry of Reconstruction and the Badische Anilin- und Soda-fabrik for the exploitation of the company's process of manufacturing synthetic ammonia and derivatives thereof (cf. J., 1920, 305 B). The directorate of the new company includes representatives of various banks, the Cie. des Mines de Béthune, the Cie. Nationale des Matières Colorantes et de Produits Chimiques, the Etablissements Kuhlmann, the Société des Produits Azotés, and Solvay et Cie. (Brussels).—(Rev. Prod. Chim., Jan. 31, Feb. 15, 1921.)

The Upper Alsatian Potash Mines (Société des Mines de Potasse du Haut-Rhin, Société par actions à Mulhouse) has just formed a subsidiary company with a capital of 1,500,000 fr. to exploit the potash mines in Alsace, which will endeavour to acquire all mining concessions, mines or works, whether belonging to companies, private persons of the State. The original capital is 1,500,000 francs.—(Deutsch. Bergwerks-Z., Feb. 19, 1921.)

**United States.**—It is announced that the Society of Chemical Industry in Basle has decided to enlarge the Ciba Co., Inc., and to take over the whole organisation and branches of the Aniline Dyes and Chemicals, Inc. The Ciba Co. will distribute both the Swiss and American dyes. The factories owned by the Society of Chemical Industry in Basle are situated at Basle, Petite Huningue and Monthey in Switzerland, at St. Fons, in France, Fabianice in Poland, Moscow in Russia, Manchester in England, and Cincinnati in the United States.—(Oil, Paint and Drug Rep., Feb. 7, 1921.)

## REVIEWS.

## INTRODUCTION TO QUALITATIVE CHEMICAL ANALYSIS.

By TH. W. FRESenius. Seventeenth edition of the original work by C. R. FRESenius. Translated by C. A. MITCHELL. Pp. 954. (London: J. and A. Churchill. 1921.) Price 36s. net.

The appearance of an English translation of the seventeenth German edition of Fresenius's "Qualitative Analysis" will be welcomed by all English-speaking chemists. The present volume is twice the size of its predecessor, published in 1897, and there is much alteration in detail, although the same general plan is maintained. Part I deals with chemical principles and methods and the behaviour of substances towards reagents. Here are brief accounts of physico-chemical theory as it affects analysis and of the operations of the laboratory. Then follow the reactions of cations and anions, including those of the rare elements and various organic acids. Part II is concerned with the systematic course of analysis and embraces practical methods of the general course and for special cases, together with explanatory notes. Lastly, the appendix includes the reactions of alkalis and glucosides, remarks on the choice of exercises, the method of tabulating results, and solubility tables. There is an adequate index.

The guiding principle of the book is denoted by these words from the preface:—"Chemical analysis demands a knowledge of the fundamental laws of general chemistry, of analytical operations, and of the behaviour of individual substances towards reagents. Given these essentials, it is possible, with the aid of a systematic procedure, to undertake the investigation of unknown substances." The whole volume is an elaboration on a most extensive scale of this fundamental thesis.

Perhaps the analyst will find the book too large to be a bench companion, but in this case he will desire to have it near for reference, because it is a chemical classic which has been brought thoroughly up to date. Especially noteworthy is the information regarding the chemical reactions of the rare metals; probably in no other place can this information be obtained in so convenient and extensive a form. An improvement on former editions is the inclusion of tables of separation for the successive groups of metals.

A few comments on details may be made. The use of iron is quite the simplest method of separating antimony from tin, and the beginner can hardly fail to succeed with it; but most of the acid used to dissolve the two sulphides should be removed or neutralised to facilitate the reduction of the antimonious solution by the iron. The method of separating calcium from strontium by ferrocyanide is interesting and little known. The statement (p. 630) that permanganate may be formed when a manganese compound is fused with potassium chlorate and sodium carbonate cannot pass; manganate, not permanganate, is the stable salt under

these conditions; indeed, potassium permanganate is converted into manganate when fused with alkali carbonate even in presence of chlorate.

On pp. 634-5 it is said that the ammonium sulphide precipitate may possibly contain, not only hydroxides and sulphides of various metals, but also phosphates, borates, oxalates, silicates, and fluorides; and there follows a very elaborate scheme for the separation of all these components. It is unnecessary for this precipitate to contain any of the latter salts, and the elaboration may consequently be avoided.

By various means interfering anions can be eliminated from solution before testing for kations, just as interfering kations are eliminated, by means of sodium carbonate, before testing for anions.

On p. 113 the compounds  $\text{HAuCl}_4$  and  $\text{H}_2\text{PtCl}_6$  are wrongly called aurohydrochloric acid and platinumhydrochloric acid respectively; and on p. 199 the salts of trivalent titanium are unfortunately called titanic salts.

The translator and publishers are to be congratulated on their work. The style and arrangement leave nothing to be desired. It is necessary, however, to call attention to one blemish, viz., that some of the formulæ are misprinted. In the table above referred to (pp. 634-5) the following erroneous formulæ occur:  $\text{H}_2\text{B}_2\text{O}_7$ ,  $\text{K}_2\text{Co}(\text{NO}_2)_6$ ,  $\text{CaF}_2$ ,  $\text{Ba}_3(\text{PO}_4)_2$ ,  $\text{Mg}(\text{NH}_4)\text{PO}_4$ ; and there are a few other similar errors elsewhere. Small defects like the above, however, scarcely detract from the value of so great a work.

R. M. CAVEN.

**CHEMISTRY OF PULP AND PAPER MAKING.** By E. SUTERMEISTER. Pp. 479, with 55 text-figures and 31 plates. (New York: John Wiley and Sons; London: Chapman and Hall. 1920.) Price 33s. net.

This volume has been prepared "with the idea of helping the young technical man, whether chemist or chemical engineer," in the pulp and paper industry. The author is a well-known mill chemist of some twenty years' experience, and the book is based on his "personal notes and experiences" collected during that period.

As a scientific introduction to the chemistry of the industry it falls below the level of Griffin and Little's "Chemistry of Paper Making" (now old and out of print), and as a manual of technology cannot be classed with Cross and Bevan's "Text Book of Paper Making" (fifth edition, 1920). We make this comparison because in our opinion these are the two text-books which have had the most influence on the industry in America and England, and also because the general arrangement and aim of the book now before us follow on the lines laid down in these "classics" of paper-making.

On the whole, this book may be fairly described as a mill chemist's note-book, based on a limited but practical experience of the industry, arranged on a well-conceived plan, and enlarged by copious references to current literature on the subject. Its chief points of interest to the paper-mill chemist in this country are:—(1) An excellent and concise account of American woods and wood-pulp processes (comprising about one-third of the volume); (2) about two dozen well-reproduced half-tone plates of paper-making fibres (from photomicrographs); (3) the numerous references (over 300) to technical literature given in the foot-notes; and (4) indications of American practice in certain routine tests from which some very useful hints may be gathered by the practical man who has sufficient experience to read between the lines.

The author writes in a simple and clear style, but we do not consider his habit of stating inconsistent conclusions suitable to the young student. For example, his claim (p. 250) that bleached sul-

phite possesses greater strength than the unbleached fibre, compared with his statement (p. 252) that the more bleach used the greater will be the attack on the cellulose, is confusing. The first paragraph on rosin (p. 264) contains an obvious mis-statement concerning reasons for selection of the grades used in sizing.

From the consulting chemist's point of view this volume is a commentary on a certain aspect of the technical chemist's work. It is neither pure nor practical chemistry, and applied chemistry is only incidental and explanatory. The successful paper-mill "chemist" is expected to have a knowledge of practical botany, mineralogy, engineering, and physics in addition to chemistry, and his chemical analyses are done largely to interpret and confirm the results of physical tests.

The author presents much valuable information in a readable and handsome form, but we do not consider that he succeeds in attaining fully the aims outlined in the title and preface.

JAMES STRACHAN.

**THE PRACTICE OF LUBRICATION.** By T. C. THOMSEN. Pp. x. + 607. (New York and London: McGraw-Hill Book Co., Inc. 1920.) Price 36s. net.

Mr. Thomsen in his preface rightly states that lubrication has for many years received only scant attention. Surely this was inevitable so long as lubricating oil companies regarded the scientist as an unnecessary adjunct to their advancement. Now that the engineer is demanding more exact information about the oil he uses, there is a growing feeling that more chemists are needed to investigate the many problems presented. Lately several well-known workers in the industry have written books to meet the growing demand for knowledge, and Mr. Thomsen is one of them. Mr. Thomsen's opportunities for acquiring information on lubricating systems have probably been unique; therefore, a record of his experiences is very welcome.

The book is designed chiefly for the engineer, but since the chemist is called upon to understand lubricating systems, it must also appeal to the chemist. The engineering sections are really well done, and the illustrations are very clear. The chemical portion is scanty, but doubtless this was deliberate, as the main object of the book was to deal with the actual use and application of lubricants in practice. There is just a hint, however, in the preface that the chemist is merely an assistant to the engineer!

From time to time various theories have been advanced to explain the production of acids and of solid deposits from mineral oil, and perhaps the belief that the unsaturated hydrocarbons are the progenitors of these decomposition products has been the most popular. The theory appears to have been based upon speculative arguments and not upon experimental results. Thomsen's faith in this theory appears to be evident from the fact that he states that hydrocarbons of the formulæ  $\text{C}_n\text{H}_{2n-2}$  and  $\text{C}_n\text{H}_{2n-4}$  are more or less unsaturated; in fact the former group is characterised by the acetylene linkage. Further, he states that the colour of an oil is due to the presence of unsaturated hydrocarbons, and that colour is a criterion of the stability of an oil. It does not require much experimental work to shed considerable doubt upon the accuracy of these statements.

One would prefer to have seen the word "adsorbed" instead of "absorbed" on page 9; and on page 14 it would have been better to state that nitronaphthalene masks the fluorescence instead of removes it. The apparatus described on p. 591 was surely designed at the National Physical Laboratory, and that which Thomsen suggested and described in the J. Inst. Elect. Eng., 50 (1915), 239,

155, is quite different from this. In this connexion it might be suggested that a few references to other workers would enhance the value of the book.

The large number of oil specifications are of great value, although in some cases differences of opinion may be expected. The inclusion of the subject of oil consumption is also very valuable, but no doubt the reader will be astonished at the wide limits given; but these are inevitable owing to the different conditions prevailing in engines of even the same class.

Whenever a chemist is called up to report upon a lubricant, he will benefit by referring to "The Practice of Lubrication" before appending his signature.

E. A. EVANS.

**BIBLIOTHECA CHEMICO-MATHEMATICA.** *Catalogue of Works in Many Tongues on Exact and Applied Science, with a Subject-Index. Compiled and Annotated by H. Z. and H. C. S. Two volumes. Pp. xii+964. (London: Henry Sotheran and Co. 1921.) Price 63s. net.*

These volumes are of so unusual a nature that they are worthy of detailed notice. The main contents of the work were first issued as a series of annotated sale catalogues, and in their original form they were of considerable value to bibliographers and librarians for reference purposes. Their publication in two well-bound volumes, together with an adequate subject index, should increase the scope of their usefulness. The addition of portraits and facsimiles of high artistic and technical merit adds to their attractive character. The portrait of Dr. Thomas Young is a particularly fine example of photo-reproduction. The biographical and descriptive references are both interesting and instructive, though it is a pity that extraneous comments were introduced in the last few pages published during the war. We may agree with the sentiments, but the remarks hardly appear to have any direct connexion with the items to which they are attached.

In view of the large field covered by these volumes, it is not remarkable that a few inaccuracies occur. On page 208 there is a statement that only one volume was published by the Royal College of Chemistry, that of 1845-7, whereas a second was published in 1853, covering the years 1848-51. That Storer's Dictionary, Cambridge (Mass.), is "the only dictionary of chemical solubilities ever attempted" is also erroneous, as one was compiled by Comey in 1896, and another by Seidel in 1907 (2nd edition 1920). The first edition of Accum's "Chemical Amusement" is dated 1819, but as the copy contains a list of chemical apparatus not included in the 1819 edition, this is obviously a misprint for 1817.

The mention of a "neatly written" MS. as possibly the one used by Robison when compiling his edition of Black's Lectures suggests the speculation as to how many of these manuscript copies are still in existence. From time to time the writer has come across these MSS., the owners of which fondly supposed that their copy was either the original, or that of Robison. Reference to Robison's preface shows that the former consisted of single octavo leaves, full of erasures and difficult of transcription, whilst the latter contained whole pages and many alterations in Black's autograph, so that either should be easily identified.

Messrs. Sotheran are to be congratulated on their enterprise in producing a catalogue of books which is also a record of permanent utility, and by publishing it in its present form they have rendered a distinct service to those interested in the history and bibliography of science.

F. W. CLIFFORD.

## OBITUARY.

S. H. BLICHFELDT.

By the death of Sören Høy Blichfeldt at the early age of 44, the margarine industry loses one of its ablest technologists. Blichfeldt, who was Danish by birth, graduated from the Royal Pharmaceutical College, Copenhagen, and came to England in 1906 to take up the position of technologist to the Maypole Works at Southall. He had previously been engaged for a few years on bacteriological and fermentation research work, and having a remarkable gift of applying science in the works he was able very soon to improve and develop methods of manufacture. This service was rendered not merely in the narrower sphere of his company, but in the wider interests of the margarine industry generally.

S. H. Blichfeldt's name will be familiar to members as a contributor to the Society's literature, and one could not meet him without being impressed by the great fertility of his mind and his wonderful power of conceiving methods and processes. Many men with faculties so fully developed are so absorbed in their theories that all else in life is excluded; but Blichfeldt was a keen sportsman, a skilled musician and, to all who knew him, a charming companion—a wonderful combination which makes his loss the more keenly felt by his colleagues and friends.

E. SKJOLD.

## PUBLICATIONS RECEIVED.

**APPLIED COLLOID CHEMISTRY: GENERAL THEORY.** By W. D. BANCROFT. *International Chemical Series, edited by H. P. TALAOT.* Pp. 345. (New York and London: McGraw-Hill Book Co., Inc. 1921.) Price 18s.

**ELECTROLYTIC DEPOSITION AND HYDROMETALLURGY OF ZINC.** By O. C. RALSTON. Pp. 201. (New York and London: McGraw-Hill Book Co., Inc. 1921.) Price 18s.

**LEHRBUCH DER FARBENCHEMIE.** By Dr. H. T. BUCHERER. *Second edition, revised.* Pp. 636. (Leipzig: Otto Spamer. 1921.) Prices: unbound 47s. 1d., bound 51s.

**A TEXT-BOOK OF DYE CHEMISTRY.** By G. VON GEORGEVICS and E. GRANDMOUGIN. *Second English edition, translated from the fourth German edition (with additions) and revised by F. A. MASON.* Pp. 560. (London: Scott, Greenwood and Son. 1920.) Price 30s.

**THE CHEMISTRY OF SYNTHETIC DRUGS.** By P. MAY. *Third edition, revised.* Pp. 248. (London: Longmans, Green and Co. 1921.) Price 12s. 6d.

**RED-LEAD AND HOW TO USE IT IN PAINT.** By A. H. SAGIN. *Third edition, rewritten and enlarged.* Pp. 139. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall. 1920.) Price 11s. 6d.

**LEAD ORES.** By T. C. F. HALL. *Imperial Institute Monographs on Mineral Resources, with special reference to the British Empire.* Pp. 127. (London: John Murray. 1921.) Price 6s.

**TRANSACTIONS OF THE INCORPORATED SANITARY ASSOCIATION OF SCOTLAND, 1920.** (Published by the Association, 88, Bath Street, Glasgow.)

**JOURNAL OF INDIAN INDUSTRIES AND LABOUR.** Vol. I., Part I. February, 1921. (Calcutta: Superintendent Government Printing. 1921.) Price Rs. 1-8 per part, Rs. 4-8 per volume of 4 parts.

**CORRIGENDUM.**—In the last issue, p. 97 R, col. ii., in the sixth line from top, read "geraniol" instead of "geraniol."



## SOCIETY OF CHEMICAL INDUSTRY.

## THE ANNUAL MEETING, 1921.

## MESSAGE FROM THE PRESIDENT.

As has already been announced, the next annual meeting of the Society of Chemical Industry will be held in Canada, in response to a very hearty invitation extended by the Montreal Section; the formal meeting extends from August 29 to August 31, but our Canadian friends have expressed the wish that the party from Europe will arrive a few days earlier so that they may be shown some parts of Canada not included in the extensive official programme now being compiled. It will thus be convenient if as many of the visitors as possible will travel with me by the "Melita," which leaves Liverpool on Friday, August 12, and arrives at Montreal on or after August 22.

At the close of the business meeting on August 31, the members will leave Montreal by a special train for a four days' triangular tour of Eastern Canada, during which visits will be paid to the paper and pulp works at Grand Mere, the largest in Canada, to the carbide and synthetic-acetic-acid factories at Shawinigan, and possibly to the aluminium and magnesium electrolytic plants. September 2 will be spent at Ottawa and the next two days at Toronto. A boat trip across the Lake has been arranged in connexion with a visit to Niagara and to the chemical works and fruit farms on the Canadian side of the river. September 6 will be spent in an inspection of the electrochemical works on the American side of the Niagara Falls, and the party will then proceed by train to New York.

From September 7 to September 10 the party will co-operate in a joint meeting of the Society of Chemical Industry and its American Section and the American Chemical Society; it will visit the great Chemical Exposition which opens at New York on September 12, and which will certainly be one of the most important exhibitions of the chemical industry which has ever been held.

During the critical period through which all branches of the chemical industry are at present passing, when old markets are falling away and must be replaced by new ones, it is more than ever essential that British industrial chemists should seize every opportunity of gaining acquaintance with the conditions which prevail in the British Dominions and abroad; only by personal visits and by the establishment of new friendships and fresh business relationships can our members hope to be able to raise our chemical industries to the position indicated for them by the history and the geographical environment of these islands. Our Canadian and American friends have intimated their desire to furnish uncommonly extensive facilities for the inspection of works concerned with every branch of chemical industry, and it is certain that similar facilities cannot be granted to private individuals travelling alone apart from an official party such as is now being formed. The Council therefore desires to urge the members of our Society to make every effort to accept the generous invitation extended to us by our transatlantic friends; we are convinced that the maximum benefit to our Society and to the chemical industries of Great Britain, of Canada, and of the United States can only be derived by the attendance of a large British party at the coming annual meeting.

WILLIAM J. POPE.

Supplementary to the provisional programme which appeared in the *Journal* for December 15 last, we are now able to give the following details

concerning the arrangements which have been made for the period August 26-31, to be spent at Montreal:—

Friday and Saturday, August 26-27:—Reception Committee meets Visitors.

Sunday, August 28:—Golf. Motor-trip around City and Island.

Monday, August 29:—

Morning: 10 a.m., Council Meeting at McGill University; 11 a.m., Annual Meeting. (a) Address of Welcome; (b) Reply; (c) General Business of the Society.

Afternoon: 1 p.m., Civic Reception and Lunch. 3 p.m., Special Visit to McDonald Agricultural College. Supper and Garden Party.

Tuesday, August 30:—

Morning: 10.30 a.m., (a) Address by a prominent Canadian chemist; (b) Pulp and Paper Symposium (3 papers).

Afternoon: 1 p.m., Luncheon at Windsor Hotel, given by Montreal Section. 3 p.m., Visits to various industrial plants.

Evening: 7.30 p.m., Banquet at Windsor Hotel.

Wednesday, August 31:—

Morning: 10 a.m., Special Addresses and Technical Papers.

Afternoon: 1 p.m., Luncheon at Windsor Hotel given by Montreal Section. 3 p.m., Special Convocation at McGill University. 4 p.m., Trip down Lachine Rapids.

Evening: 11.55 p.m., Leave by night train for Shawinigan.

## NEWS FROM THE SECTIONS.

## SHAWINIGAN FALLS.

At a meeting held on March 12, Dr. F. W. Skirrow presiding, Dr. John S. Bates, formerly director of the Forest Products Laboratories of Canada, gave an address on "Pulp and Paper."

The subject of forestry should be taken more seriously by the public, because the time has come when plans must be carried out on a large scale to protect and reproduce our crop of trees for all the wood-using industries. Canada's reserves of timber rank next to those of Russia and the United States, but even this strong position must be protected to ensure permanent supplies for the Dominion and the rest of the world. Forest fires have burned down ten times as many trees as the loggers have felled. In the east there are still large supplies of pulpwood trees, but British Columbia has most of the big timber. Fortunately, over 90 per cent. of the timber land remains under Government ownership, and the problem is fairly simple if the public will only demand and assist State action in conserving the timber resources. As over two-thirds of the land area in the settled portion of Canada is unfit for agriculture, there is ample scope for development.

The pulp and paper industry now leads the list of manufactures in Canada, and also leads all others in export trade. This may be traced directly to the natural resources of pulpwood and water power. To a large extent paper is made from raw materials not suitable for other purposes. Cotton goes into textiles, and the waste clippings, rags and linters supply the higher-grade paper mills. The best trees are cut into lumber, but the smaller sizes and inferior species are good enough for most grades of paper products. The problem in making paper pulp is to dissolve out the lignin and other carbohydrates in the wood, and to separate the flexible fibres of white cellulose. Wood is the outstanding source of fibre for papermaking, because the length of the natural fibre is just right, the yields of pulp are high, wood is cheap, abundant and compact, and, finally, it floats down our rivers



and is easily handled. The conifers such as spruce, balsam fir, hemlock, and jack pine are low in resin content, and have fibres about  $\frac{1}{4}$  in. long and 0.01 in. wide. The hardwoods such as poplar, bass-wood, and birch are more resistant to pulping, and have fibres about one-third the size of the soft-woods, so that their use is more limited.

The speaker then outlined the groundwood, sulphite, sulphate, and soda processes by which wood is converted into mechanical and chemical pulp. The characteristic properties of the different kinds of pulp fibres were illustrated by samples of paper pie-plates, newsprint, wrapping paper, board products, magazine paper, writing paper, and bank-note paper. The methods of beating, bleaching, sizing, and loading to give the desired formation and finish were described. In conclusion, the scientific trend of the industry in the direction of improvements in processes and machinery, saving of by-products, and quality of products was pointed out.

#### BRISTOL AND SOUTH WALES.

A meeting was held at the University College, Cardiff, on March 4, with Prof. C. M. Thompson in the chair. Mr. Donald Brown read a paper on "Paper-making," in which he described the various processes employed from the felling of the timber in Sweden to the finished roll, and explained the different types of machinery used. The paper had special reference to the Ely Paper Mills, Cardiff, which were subsequently visited by the Section on March 10, by the courtesy of Messrs. T. Owen and Co., Ltd.

At Bristol on March 17, Mr. E. Walls (chairman) gave an address on "Chemical Industry in Bristol."

Except for the purely engineering, boot manufacturing and clothing trades, there was no productive industry in Bristol which was not interested in technological chemistry. Chemical engineering was strongly represented in gas manufacture, an industry which was introduced into their city in 1811 by a Mr. Briellat, who was accused of having "unholy dealings with an infernal power." The Bristol Gas Co. started operations in 1816 with a capital of £5000 and experienced many vicissitudes (among which was the refusal to lay pipes on account of the danger), but it outlived the competition of a works which started producing oil gas in 1824. The local tanning industry derived much benefit from the importation of hides from Ireland, and the oil, paint, and varnish industry probably owed its origin to local supplies of ochre and importation of logwood from the Indies. Floorcloth, the forerunner of linoleum, was introduced in Bristol by John Hare in 1782. The fame of Bristol brewing went back many centuries. The distillation of petroleum for toluene was carried out on an enormous scale during the war; in fact, no less than 80 per cent. of the total output of toluene in 1915 was produced at Portishead, the plant for which was shipped *en bloc* from Rotterdam. Alcohol was distilled locally for munitions purposes during the war, as were also aromatic hydrocarbons, phenol, and glycerin. At Netham there was the large plant of the United Alkali Co. producing soda by the Leblanc process, also caustic soda, chlorine, bleaching powder, sulphuric acid and other products. Glass-making was an old-established Bristol industry largely engaged in making bottles for imported wine; in 1797 there were 14 glass works in operation. The Avonmouth works, erected during the war, might have given Bristol a preponderating position in chemical industry, the total capacity of the sulphuric acid plant being sufficient to supply the whole requirements of Europe, but, unfortunately, it had been closed down. In fine chemicals they had no standing, but Bristol had always been a pharmaceutical centre. The refin-

ing of copper and manufacture of brass from cupriferous residues from Cornish tin-ore was started about 1705, calamine from the Mendip mines being also used. The brass-wire industry was developed on a large scale, and in 1712 it was stated that the brass and copper works consumed 2400 tons of coal per week. There was also an important lead industry in the 17th and 18th centuries. The oldest chemical industry in Bristol was soap manufacture, which dated back to the 13th or 14th century; this industry suffered severely in 1681 when the King granted the monopoly of soap-making to certain Londoners. In conclusion, Mr. Walls referred to the help which the local chemical industries and the Section had received from the Chemical Department of the University, and stated that with such assistance there should be a bright future for chemical industry in Bristol.

#### GLASGOW.

At an informal meeting of the Section held on March 9, Mr. J. H. Young presiding, Mr. Crooks read a paper on the "Electric Charge on Colloids." Reference was made to the importance of Colloid Chemistry and the growing necessity for all technical chemists to have a sound knowledge of the subject in view of the fact that many of the processes which the chemist controls deal with colloidal phenomena. The work of Graham was mentioned, and emphasis was laid on the fact that the term "colloid" connotes a "state" and not a "form" of matter, and that many of Graham's crystalloids have, by various methods, been obtained in the colloidal state.

Malfitano's work on the conductivity of sols after filtration through a collodion membrane was referred to, but it was pointed out that his method was open to objection as the filtration might change the concentration of the medium as well as remove the disperse phase. The method of prolonged electrolysis used by Whitney and Blake was recommended as a more suitable method. The necessary precautions to be taken in observing migration velocities were fully discussed. The action of electrolytes on sols was described and the "Valeoccy Rule" generalisation enunciated and explained. Burton's estimation of the charge on a colloid particle (by assuming that the amount of aluminium ion necessary to precipitate a certain amount of a sol carried the same amount of positive electricity as the precipitated colloid did of negative) was also dealt with. In concluding, reference was made to the theories regarding the electrical charge, particular attention being given to the "differential diffusion" explanation of Taylor, the more or less generally accepted "adsorption" explanation, and Burton and Hardy's explanation for the oxidisable and non-oxidisable metals.

A paper on "Lead Corrosion" was read by Mr. W. G. McKellar.

#### NEWCASTLE.

Two papers were read at the meeting held on March 23. Mr. C. F. Tidman re-read the paper on "Alcohol from Coke-oven Gas," which he had previously read at Middlesbrough on March 7 (*cf.* J., 1921, 103 a), and Mr. C. L. Haddon contributed a paper on "The Effect of Some Physical Conditions on Calcium Sulphate Cement." Prof. P. Phillips Bedson presided.

Mr. Haddon has carried out tests on the expansion of a flooring plaster consisting of 3 parts of plaster to 1 part of water or of a 5 per cent. solution of calcium chloride, potassium sulphate, or ferrous sulphate. Le Chatelier's apparatus was used, and no measurements were made with the cement in a plastic state. He found that expansion was least with accelerating solutions and

greatest when retarders were employed. With the potassium sulphate solution, wet-set plaster continued to expand during recrystallisation. Both the degree of wetness and the amount of expansion have an effect on the tensile strength; briquettes which had expanded 5 per cent. had less than a quarter of the strength of similar briquettes allowed to dry in air (when the expansion is very much less). Under normal conditions, expansion does not exceed about 0.1 per cent., and ceases within the time required for complete setting.

The weakening of flooring plaster was investigated by immersing briquettes (after six hours' immersion in water and air-drying) in various liquids for definite times and then breaking them. It was found that mineral oils were without effect, but the tensile strength was reduced by mixtures of methylated spirit and water to an extent varying with the amount of water in the mixture. The strength was reduced by over one-half by water and by a concentrated solution of calcium sulphate. Similar results were found for crushing strengths.

The value of the published data on the mechanical strength of flooring plaster is impaired by the circumstance that no standard has been laid down concerning the condition of the briquette on breaking, and it is suggested that the method used in testing Portland cement should be generally adopted, and that the proportion of flooring plaster to water should be 3:1 by weight.

#### MANCHESTER.

The annual meeting was held at the Textile Institute, Manchester, on April 1, Mr. John Allan presiding. Five members of the committee retired, and as there were seven nominations to fill the five vacancies a ballot was taken; Messrs. R. H. Clayton, J. R. Hannay, W. B. Hart, J. A. R. Henderson, and J. Huebner were elected.

The Hon. Secretary, Mr. L. Guy Radcliffe, then read his report for the session 1919-21, which stated that the original syllabus, issued last August, arranging for eight meetings and about twelve papers, had been carried out with very few alterations. The change of meeting-place from the Grand Hotel to the Textile Institute, which was decided upon by a unanimous vote of the members present at the February meeting, has had the success anticipated, and the Section has now the advantage of an excellent locality for scientific meetings and social intercourse. The numerical strength of the Section has diminished in spite of many new members having joined the Society. About 20 members were lost by resignations, and 5 by deaths, but removals from the district were very numerous, and the membership now stands at about 620. The committee has elected Dr. E. Arden to be chairman in place of Mr. John Allan, who now becomes vice-chairman in succession to Mr. W. Thomson, and the hon. secretary was re-elected for a further year. The report also refers to the annual dinner held in November and the annual supper held in March.

At the ordinary monthly meeting which followed, Mr. H. J. Bailey, formerly of the Explosives Department of the Ministry of Munitions, read a statistical paper on "Sulphuric Acid during the War Period." It was shown that whereas the production of sulphuric acid in the United Kingdom is normally about one million tons per annum of 100 per cent. strength, 1,208,000 t. was produced during 1916 in chamber-acid plants; and that the production of concentrated or fuming sulphuric acid made in contact-acid plants reached 221,000 t. during the year 1917.

The raw materials for the manufacture of sulphuric acid were dealt with, and tables were given showing the various main uses in the heavy chemical

industries of the country. In addition, summarised statistics were given of the consumption of sulphuric acid in the manufacture of the various types of explosives, and it was shown that in the period 1916 to 1918 the overall consumption per lb. of explosive manufactured was reduced to less than one-half of that used during 1915 by obtaining increased efficiencies and necessary economies in working.

The author also discussed the production and use of fertilisers during the stressful period of the submarine menace in 1917-18, and showed that whereas during the first two years of the war the manufacture had seriously declined, before the war had ended the full pre-war production had been surpassed, thus enabling much larger supplies of sulphate of ammonia to be available for agriculture.

#### LONDON.

At a meeting held on April 4 at Burlington House, with Mr. Julian J. Baker in the chair, a paper entitled "The Scientific Principles of Cold Storage" was read by Prof. W. Stiles.

Food materials that can be preserved by cold storage—and they are the majority—may be roughly divided into:—(a) Those, such as fruit, which deteriorate on freezing and therefore must not be cooled below 0° C.; and (b) those, such as mutton and rabbits, which, after being frozen solid, practically revert to their original condition on thawing. A few materials, *e.g.*, beef and some species of fish, occupy an intermediate position. Cold storage above 0° C. allows enzyme and other actions, which may lead to deterioration, to continue, but below this temperature these processes are stopped or greatly retarded. Further advantages of frozen storage are:—Longer preservation, the possibility of close packing, and the small amount of supervision required. One of the greatest problems of cold storage is therefore to devise means by which materials belonging to class (a) may be stored in a frozen condition instead of at temperatures above 0° C.

The cold storage of fruit is influenced by variation in the rate of cooling, light, relative humidity, movement of the air, and other factors. Even at 0° C. biochemical changes take place which result in the conversion of carbohydrates and organic acids into other substances, and the production of carbon dioxide by respiration. The rate of respiration is greatest in those fruits that keep in cold storage for the shortest time, and this rate is doubled for an increase of temperature of 8° C. Although chemical changes are reduced by cooling, all reactions are not depressed to the same extent. Fruit may be chilled slightly below 0° C. without freezing, owing partly to the lowering of the freezing point by the salts etc. present in the juices, and partly to the effect of the small amount of heat produced by respiration. When rotting commences the temperature of the fruit increases rapidly. In storage by freezing, the change from liquid to solid is not completely reversible on thawing, and with beef and fish, water separates which runs out on thawing, thus leading to loss of soluble substances and probably causing toughness. The rate of cooling much affects the condition after thawing, and experiment has proved that with rapid chilling the changes produced are reversible on subsequent thawing. Although agitation of the cooling medium has little effect upon the rate of cooling, this property is largely influenced by the conductivity for heat of the medium. Gases are unsatisfactory, and the most efficient practicable media are water and aqueous solutions of common salt, calcium chloride or magnesium chloride. Materials of small bulk, such as fish or small joints of meat, are very effectively frozen in brine, but with larger pieces the outer layers are often dull in

colour, although the salt present is insufficient to affect the flavour after cooking. Calcium and magnesium chlorides may give rise to a bitter flavour. But little experimental work has so far been done in this subject, and its extension would prove of the greatest value to all civilised races.

### JAPANESE CHEMICAL INDUSTRIES FROM THE STANDPOINT OF FOREIGN TRADE.

As is well known, the chemical and allied industries of Japan underwent a sudden and great development during the war, owing mainly to the country being cut off from extraneous sources of supply, but also to the great demand for warlike materials. The progress achieved is well reflected in the following statistics of foreign trade, which afford a comparison between the average yearly trade during the three years immediately preceding the war and the trade in 1918. During this period the value of the imported chemicals, etc. rose from 63 million to 93·4 million yen, an increase of 47 per cent., and that of the exports increased from an average of 51·56 to 246·79 million yen, a gain of 378 per cent. The appended table gives some details (the negative sign indicates a decrease, and the yen is taken at 2*s.*):—

	Imports.		Exports.	
	Total inc. or dec.	Rate per cent.	Total inc. or dec.	Rate per cent.
Sugar .. ..	£216,200	356	£1,385,000	133
Leather .. ..	111,300	36	940,000	753
Oils and waxes ..	181,300	39	3,420,000	426
Chemicals and drugs ..	2,394,600	227	2,656,900	474
Explosives .. ..	160,900	183	1,708,200	150
Dyestuffs .. ..	417,000	58	—	—
Pigments .. ..	129,900	95	—	—
Paints .. ..	27,400	16	1,024,800	2601
Rubber .. ..	-104,600	-42	—	—
Celluloid .. ..	-426,000	-100	—	—
Photographic materials	101,800	104	—	—
Paper .. ..	502,900	45	2,654,100	857
Porcelain .. ..	-1,800	-18	1,413,500	242
Glass .. ..	-134,600	-42	1,296,000	416
Enamelled ware ..	-3,800	-100	—	—
Bricks .. ..	-400	-2	—	—
Cement .. ..	—	—	560,200	1376
Electric-furnace pro- ducts .. ..	117,400	970	—	—
Fertilisers .. ..	-1,269,200	-98	21,900	32
Total .. ..	£2,935,400	47%	£19,522,600	376%

It will be observed that very remarkable increases occurred in the imports of chemicals and drugs, dyestuffs, paper, sugar, oil and wax, leather, explosives, pigments, photographic materials, and electric-furnace products. On the other hand, celluloid, enamelled ware, and fertilisers all showed noteworthy decreases. Of the chemicals, very large quantities of soda ash, caustic soda and soap were imported, although production has increased to such an extent that at the present time Japan can supply 80 per cent. of her consumption of caustic soda from home sources. The increases shown are to a very large extent due to the enormous rise in market prices; the quantities imported actually diminished.

The following figures show the percentage value of the imported chemical products compared with the total value of the whole import trade of the country:—

	Average of 3 pre-war years.	1918.
(a) Total imports .. ..	£61,990,500	£165,814,200
(b) Technical products ..	—	57,386,700
(c) Chemical products ..	6,354,900	9,340,800
Ratio of (c) to (a) ..	10.1%	5.6%

The largest increase in the value of exported products was obtained by the oil and wax industry,

mainly as a result of the new export trade in palm oil, rapeseed oil, and soap. The chief increases in chemicals and drugs were in calcium carbide, acetic acid, and potassium chlorate. The ratio of the average value of the chemicals, etc. exported before the war to the whole export trade rose from 9·6 to 12·2 per cent.:—

	Average of 3 pre-war years.	1918.
(a) Total exports .. ..	£53,552,500	£106,210,000
(b) Technical products ..	—	£172,890,000
(c) Chemical products ..	5,156,500	24,679,400
Ratio of (c) to (b) ..	—	14.1%
Ratio of (c) to (a) ..	9.5%	12.2%

The most important countries of destination of exported Japanese chemical products are China, India, Oceania, and South America.

### THE INTERNATIONAL NORMAL WEIGHT FOR THE SACCHARIMETER.

At the present time the normal weight that is in almost universal use for the saccharimeter (or polarimeter reading the percentage of sugar directly) is 26·0 grams, this standard having been adopted by the International Commission for Uniform Methods of Sugar Analysis in 1900. On dissolving 26·0 grams of pure sucrose in water, and making the liquid up to 100 metric c.c. at 20° C., a solution is obtained which in a 200-mm. tube reads exactly 100·0 scale divisions corresponding with 34·657±0·023 angular degrees with spectrally purified sodium light. In France a normal weight of 16·29 grams is largely used.

The proposal was, however, recently made by Dr. C. A. Browne and other American chemists to adopt a sugar scale having a normal weight of 20 grams, the principal advantages claimed being:—(1) It is a compromise between the 26·0 and 16·29 gram scales; (2) the results obtained are easily converted into percentages by multiplying by 5; (3) aliquot portions of 50, 25, 20, and 5 c.c. of the 20 per cent. solution represent even gram quantities; and (4) the specific rotation of sucrose at a concentration of 20 grams. in 100 c.c. (18·62 per cent. by weight) is about the maximum. It was further argued that the factor for conversion into circular degrees, namely 100=34·657, is inaccurate in view of the work of Bates and Jackson published in 1916, and that the present is an opportune time for adopting a new factor and a new standard. French chemists decided to support their American colleagues in adopting the 20 gram normal weight.

In order to elicit the opinion of British chemists in the matter, a committee was formed consisting of the following:—Prof. Arthur R. Ling (chairman), Prof. Thomas Gray, and Messrs. L. J. de Whalley, Hugh Main, and J. P. Ogilvie (secretary). This committee drew up a statement of the arguments *pro et contra*, nearly 2000 copies of which were sent to chemists engaged in the sugar and allied industries, both in this country and in the British Dominions.

An analysis of the replies received shows about 72 per cent. of the correspondents to be opposed to the adoption of the new standard. Most of these replies stated as the reason for the decision that the advantages claimed for the proposed new standard were too slight to compensate for the considerable inconvenience, expense, and confusion that would (it was considered) be involved by its adoption. A very frequent additional reason was that, owing to the smaller amount of sample taken for the assay, the accuracy of observation would be diminished. In an addendum to his reply, Mr. A. F.

Blake, Chief Chemist, Atlantic Sugar Refineries, Ltd., St. John, N.B., Canada, said that he thought the 20-grain value should be retained regardless of whether Herzfeld's or Bates and Jackson's factor is the correct one, or whether future investigations provide still another, "new instruments being made according to the best factor available at the time, and old ones being controlled and corrected by quartz plates standardised according to the latest factor."

It would therefore appear that British chemists are largely in favour of retaining the present international standard, which in fact is now in almost universal use (excepting in France and Mauritius).

## CHEMISTRY AT THE INDIAN SCIENCE CONGRESS.

The Eighth Science Congress was held this year from January 31 to February 5, at Calcutta, under the presidency of Sir Rajendranath Mukerjee. About 150 papers were presented.

On February 1, Dr. E. R. Watson, Director of the Cawnpore Research Institute, gave an address on "Chemical Research for the Development of Industries in India." Speaking of the dye industry, he said that the amount of coal tar at present available was quite insufficient, but if more were produced he saw no reason why the industry should not thrive. He was not very hopeful about wood distillation, but white lead, potassium permanganate, and various other chemicals might be made cheaply. The manufacture of sulphuric acid was handicapped by the want of indigenous sources of sulphur. There was some talk of making it from natural sulphates, but it was very doubtful whether this would pay. Industrial alcohol could be prepared from grain. There were three classes of industries possible in India, viz., (1) iron and steel, (2) textiles, and (3) chemicals.

In the discussion that followed, Dr. H. K. Sen, who has had considerable experience in the manufacture of sulphuric acid and sulphates in India, said that unless there were State protection chemical industries could not stand competition from outside. Sulphuric acid of sp. gr. 1.84 was sold in England at 28s. per ton before the war, but it could not be manufactured in India for less than Rs.97.5 per ton (rupee=1s. 4d. at par). Nitric acid would cost 4.5 annas (6d. at normal exchange) per lb. Magnesium sulphate used to sell in Germany at Rs.1-8 per cwt., whereas it could not be manufactured in India for less than Rs.4-14 per cwt. Dr. Sen advocated the manufacture of industrial alcohol from calcium carbide, which, according to the calculations of Drs. Simonsen and Sudborough, could be manufactured in India at £6 per ton. He said that 1 ton of carbide would yield as much alcohol as 8 tons of potatoes, viz., 700 kg. Tertiary pyritic shale was mentioned as a possible source of sulphur for the manufacture of sulphuric acid. India produced 6000 tons of saltpetre and exported 4000 tons annually. Nitric acid could be manufactured from it cheaply if the by-product potash salts could be disposed of profitably. Dr. H. E. Watson, of Bangalore, remarked that the manufacture of one or two dyes would not do; either a large number should be made or none at all. He was in favour of manufacturing industrial alcohol from cellulose by a biochemical process. It should stand competition with petrol. Prof. Gilbert Fowler, of Bangalore, expressed the opinion that the proposed manufacture of dyes in India was hopeless; nor was the manufacture of sulphuric acid from gypsum possible in India on a commercial scale on

account of the high temperature required. In Europe calcium sulphide and sulphate were waste products in the manufacture of soda, yet no one thought of making sulphuric acid from them. Industrial development must grow side by side with the solution of the food problem, which was of the utmost importance at the present time.

In the Section of Chemistry some two dozen papers on the pure or applied science were read or taken as read. The president, Dr. H. E. Watson, discussed the development of Indian chemical industry in his address. Taking into consideration the vastness of the Indian Empire and the difficulties of railway communication, he was not in favour of erecting large factories in one or two places. Small factories distributed all over the country would be more suitable. He tabulated all the industries, giving figures of merit to each and taking into consideration all the conditions as to raw material, labour, transport, demand, competition, etc. He found that the salt industry took the first place, and tiles and bricks came next. Then followed iron and steel, sugar, oil, cement, essential oils, drugs, milk products, etc. For wood distillation the conditions were unfavourable. Hardening of oil for edible purposes was quite possible. The manufacture of sulphuric acid would succeed if small contact-plants were erected in different parts of India. The soda industry could not stand competition from the Brunner, Mond or Magadi Soda companies. The manufacture of bichromate was possible, as there was a great demand for it in the tanneries. Cyanamide could be produced cheaply, but it would be very difficult to induce the Indian cultivator to use it as a fertiliser.

Messrs. G. J. Fowler and D. L. Sen described an investigation carried out on behalf of the Indian Munitions Board upon the bacteria associated with rice and other cereals, in order to study the sources of infection in starch factories, the consequent loss of starch, and allied questions. The bacillus employed by Weizmann for the manufacture of acetone and butyl alcohol from starch is very resistant and survives the ordinary cooking process, but unpolished rice carries a small quantity of an alkaloid which acts as a selective antiseptic and protects the rice from bacterial attack. Marked differences were observed between the behaviour of polished and unpolished rice even after cooking, and microscopic examination showed that the polished grain carried more organisms of greater variety. Rice can be sterilised by a dry treatment with sulphur dioxide, which, however, also inhibits germination; this is an advantage in the case of grain that is to be used for the manufacture of starch, but, of course, not for seed grain. Weizmann's bacillus can be isolated by making successive sub-cultures from an original unsterilised mash of grain into a sterile mash maintained for one or two minutes at 90° C. A very fair acetone fermentation is obtained after nine sub-cultures, showing that the isolation is a selective process rather than the acquirement of new characteristics by the organism in the course of generations.

Dr. Fowler, in conjunction with Mr. K. C. Srinivasier, found that sulphur dioxide was also a good antiseptic for use in the manufacture of glue and gelatin. In this case it must be in aqueous solution, the strength of which should be about N/8, equivalent to about 0.75 gm. of sulphur per kg. of the final product. It also clarifies the material and decolorises it. A paper by G. J. Fowler and B. Bannerji on the utilisation of *megasse* (the residue left after the pressing of sugar cane, now usually used as fuel) described how by hydrolysing it with dilute sulphuric acid under pressure a solution was obtained which, after the addition of some ordinary sugar, can be fermented for the manufacture of power alcohol. After heating under pressure with

caustic soda the residual fibre can be worked up into cheap paper stock.

Another biochemical paper, by G. J. Fowler and M. Srinivasier, dealt with the indigo dye vat. Instead of hydrosulphite, the Indians add the seeds of "cassia tora" to reduce the indigo to indigo white. These seeds contain a sulphur protein and bacteria that ferment and produce hydrogen, which effects the change of the indigo. There is also a bitter substance which acts as a selective anti-septic restraining the growth of other bacteria.

## RECENT DEVELOPMENTS IN THE ELECTROLYTIC PRODUCTION OF HYDROGEN AND OXYGEN.

A. J. HALE.

Much of the plant employed up to 1913 for generating hydrogen and oxygen by the electrolysis of water was composed of cells which had been designed prior to 1903, and although some improvements in design and efficiency were introduced during the two or three years before the war, the demand of the Allies for these gases was no doubt responsible for much of the activity recently displayed in this branch of industrial chemistry.

The cells on the market today, many of which have been devised during the last six years, may be classified as bell, filter-press, or unit type. Each cell is usually capable of taking 300–600 amperes, and the electrodes are placed near together to diminish electrical resistance. Apart from the filter-press bi-polar type, the tendency has been to develop the construction of flat-diaphragm units of maximum capacity which occupy a minimum of floor-space. These single units possess the advantage over the filter-press type that any cell in need of repair may be cut out of the series without stopping the remainder, whereas in the filter-press cells the whole group must be stopped while the repair is effected.

**Bell Cells.**—Under this head may be included all non-diaphragm cells, for the predominant idea in this type is to prevent the mixing of oxygen and hydrogen by encasing the electrodes in such a manner that the discharged gases are directed upwards to their respective mains. Sometimes the construction is unnecessarily complex, as in the Varelle cell (Fr. Pat. of 1905) and Tommasini cell (U.S. Pat. of 1912), and others; but the plant of the Hydrogen, Oxygen and Plant Co., based upon Jaubert's patents of 1916, and the Churchill cell (Pat. of M. Geeraerd of 1916) are of simple construction and reasonably efficient. Each of these cells gives over 6 cb. ft. of hydrogen per kw.-hr.; the former has been put down at several French factories, and the latter is used in several English works.

The principle of the Jaubert cell is shown in Fig. 1, taken from the patent specification. The cell consists of a rectangular heat-insulated tank, in which there are five pairs of electrodes connected in parallel. Each electrode is partly covered by a steel bell, and the dimensions of the bells and electrodes are in proportion to the volumes of gases evolved at each, i.e., 2:1; but, as indicated in the diagram, the length of electrode exposed below the bell is inversely as the volume of gas evolved. Each cell is rated to take 300–600 amperes at 2.3–2.5 volts, and the purity of the evolved oxygen is 97.5–98.5 per cent. before passing through the purifiers.

The Churchill unit resembles a filter-press in construction but has no diaphragms. Each electrode plate is so constructed that the oxygen and hydrogen are prevented from mixing when dis-

charged from adjacent electrode faces by vanes of insulating material which guide the gases upwards to their respective channels. Obviously any number of plates may be screwed together to make up a battery. The advantage claimed for this cell is that the absence of an asbestos diaphragm diminishes resistance considerably, so that only 2.0 volts is needed per cell.

Non-diaphragm cells are usually equipped with gas purifiers (consisting of heated platinum spirals) in order to increase the purity of the gases up to 99.5 per cent. or more.

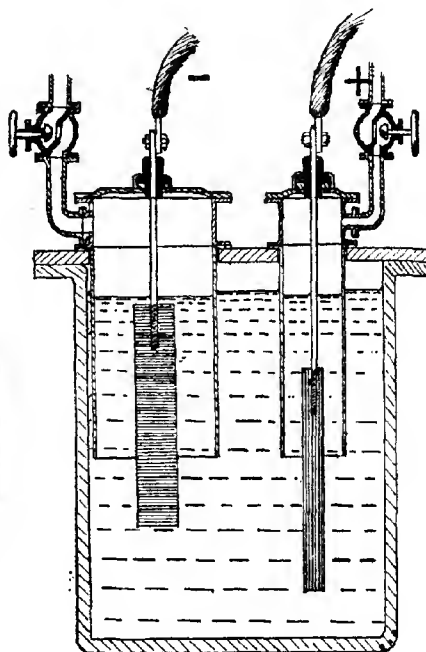


FIG. 1.

**Filter-press Cells.**—The design of this type is indicated to some extent by its name, and descriptions may be found in text-books on electro-chemistry. Several firms have put this type on the market. The National Oxygen Company of Chicago effected improvements in the filter-press design in 1917, and the existence of numerous competitors of the Oerlikon cell (Schmidt's Patents) shows that this type has many points in its favour. Some care is needed in fitting the asbestos diaphragms and in screwing together the plates before starting up, but these are minor handicaps, and such plants have been in use for many years and given entire satisfaction to the users.

**Diaphragm Unit Cells,** sometimes known as tank cells, have of late been much improved and today represent the most perfect contrivances for producing oxygen and hydrogen by electrolysis. In the early type of cell (1911) manufactured by the International Oxygen Co., an iron tank formed both the containing vessel for a caustic soda electrolyte and the negative electrode. From the lid of this vessel was suspended an iron anode corresponding in shape to the outer tank and separated from it by an asbestos sack which depended from the lid. The kw.-hr. output was 7 cb. ft. of hydrogen of 99.7 per cent. purity.

An improved form of this cell is now made by the Davis-Bournonville Co., New Jersey. The kw.-hr. output is 7.5 cb. ft. of hydrogen, and the dimensions

of a 500-ampère cell are: 13.5 in. x 24 in. x 46 in. in height; 1000-ampère units are: 15 in. x 36 in. x 51 in. in height. A gas purity of 99.5 per cent. is guaranteed, and, in addition, a speciality is made of automatic control throughout the plant which diminishes risk of accident and restricts the amount of supervision required.

The next stage in the development of the unit tank cell was reached almost simultaneously in England and America. A flat cell was designed in which the body consists of a stout steel frame of rectangular shape. On each side of the frame is clamped an iron sheet, one positive and one negative, the two being insulated from each other and separated by an asbestos-sheet diaphragm which divides the cell into two vertical halves, thus effectively preventing the mixing of the oxygen and hydrogen which are evolved when the cell is working. The cell of the Integral Oxygen Co. (Fig. 2) is of this type, and a study of its design and record of output will show that economy of floor-space and material is combined with high efficiency. Each cell covers a floor-space of 4 in. x 42 in., and the total over-all height, with piping, is about 7 ft.

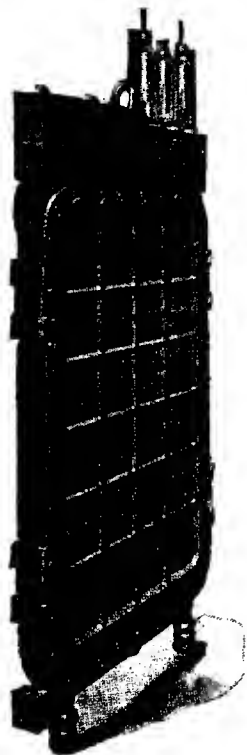


FIG. 2.

On the right side of the body shown is the nickel-plated cast-iron anode, and on the left is a similar iron cathode. The active surface of each plate is corrugated in order to give increased area and to facilitate the circulation of the electrolyte. It has been found necessary to plate the anodes of all electrolyzers with nickel in order to avoid the formation of iron-oxide deposit, which speedily interferes with the efficient running of the cell and tends to block the pores of the diaphragm. Cast as parts of the head of this cell are a reservoir for electrolyte, proper channels for collecting the gases,

and a simple arrangement of chambers and water-seals acting as an automatic internal-pressure equaliser. At the top of the diagram are shown an eye-bolt for raising and moving the cell, and three glass bells, of which the first is the hydrogen outlet, the second the feed for distilled water, and the third the oxygen outlet. These bells are protected by two iron uprights, the front one being an overflow pipe and the end one a standard, of which the function is entirely protective.

Each cell is rated at 600 ampères and 2.2 volts, and under these conditions gives 9.6 cu. ft. of hydrogen per hour. A battery of 50 cells takes up a floor space of 3 ft. 6 in. x 16 ft. 8 in., and with a current of 800 ampères at 2.4 volts will give 15,360 cu. ft. of hydrogen per 24 hours. The weight of the empty cell is 800 lb., and when filled with electrolyte 1000 lb., so that each holds about 20 gallons of water, one gallon of which will give about 160 cu. ft. of hydrogen.

The Levin cell (U.S. Pat. 1917), made by Electrolabs, Pittsburgh, Pa., is constructed on the same plan and is thoroughly efficient. A complete cell, which has the dimensions 30 in. x 25 in. x 6½ in., is rated at 250 ampères, and weighs when empty 148 lb.; a larger type, 43 in. x 37 in. x 8½ in., rated at 600 ampères, weighs 325 lb., and gives 9.6 cu. ft. of hydrogen per hour. There are three compartments, the two outer ones for generating oxygen, and the inner one for hydrogen. The three electrodes are independent of the casing of the cells, so that its construction differs in some respects from that of the cell last described.

Some idea of the efficiency of the various cells may be gained from the figures in the following table, which shows the output of hydrogen per kw.-hr. as well as the voltage needed for each cell and the guaranteed purity of the oxygen and hydrogen evolved:—

Cell.	Voltage required.	Hydrogen per kw.-hr. cu. ft.	Purity of Hydrogen Per cent.	Purity of Oxygen Per cent.
Oerlikon filter-press	2.2-2.4	6.0	99	97
National Oxygen Co., Chicago	2.0	7.4	99.5	99.3
Integral Oxygen Co.	2.2	7.3	99.5	99
Levin cell	2.2	7.3	99.8	99.5
Oxy-hydrogen and Plant Co.	2.3	6.0	98	96
Churchill Cell	2.0-2.2	6.5-7.0	99	93
Davis-Bourneville	2.1	7.5	99.5	99

*Cost and Lay-out of Plant.*—In considering the cost of purchasing and running plant for producing hydrogen and oxygen, attention must be directed to several factors. Direct current is necessary, and this may involve the installation of generating plant for converting the alternating current supply. The gases will be passed to gas holders under a pressure of a few ounces and thence, by low-pressure compressors, to storage tanks at 300 lb., and, in the event of one or both gases being sold, high-power compressors will be needed, together with steel cylinders for bottling under a pressure of about 2000 lb. per square inch.

The layout of a complete plant would consist of the cell room containing the electrolyzers, in close proximity to the generator room, if generators are needed. From here the gases would pass to the gas-holders, of the usual gasometer type, which are erected in a suitable position outside and a few yards away from the cell room, and capable of holding at least 12 hours' supply of gas. In a neighbouring house would be the compressors, one for each gas, and the compressing table for filling the cylinders used in transporting the gas to the ultimate user. If the gases are to be used on the works, low-power compressors should connect to storage tanks holding 1000—5000 cu. ft., from which pipe lines will distribute the gas to the work-benches.

The capital outlay involved may now be considered. The floor-space needed for cells to supply



5000 cb. ft. of hydrogen per 24 hours would be as follows:—

Type.	Floor-space.	Type.	Floor-space.
Integral Oxygen Co.	7'x3' 4"	Churchill.	40'x3'
National Oxygen Co.	13'x4' 6"	Hydrogen Oxygen & Plant Co.	50'x9'

The approximate cost of the electrolyzers will be £1400—£1500 in each case, and for compressors, gasholders, etc., another 150 per cent. may be added. Electrolyzers for generating 20,000 cb. ft. of hydrogen and 10,000 cb. ft. of oxygen per 24 hours will cost approximately £6000, and if the whole of the accessory plant be needed a total outlay of £15,000 may be involved.

An example showing the total cost of producing oxygen and hydrogen by this method may now be outlined, in which the costs quoted are certainly above the average.

#### Costs.

Plant to provide 5000 cb. ft. of hydrogen per 24 hours	
Initial outlay on cells, switchboard, generator, compressors and gasholders .. .. .	£4,000

#### Annual Cost of Production.

300 days of 24 hours, hydrogen .. .. .	1 500,000 cb. ft.
oxygen .. .. .	750,000 cb. ft.
Energy per 1000 cb. ft. O <sub>2</sub> } 300 B.T.U. at id. .. .. .	25s. 0d.
.. 2000 cb. ft. H <sub>2</sub> }	

#### Overhead Charges.

5% Interest on £4000 .. .. .	£200
Depreciation at 10% .. .. .	£400
Attendance, 20 hrs. per day at 2s. per hour .. .. .	£600
Annual cost of current, at id. per unit .. .. .	£925
	£2125

Cost per 1,000 cb. ft. of oxygen (hydrogen being wasted) .. 56s. 0d.

If however both gases are utilised:—	per 1000 cb. ft.
Cost of oxygen .. .. .	28s. 0d.
.. hydrogen .. .. .	14s. 0d.

Allowing for 200 cylinders at £5, i.e., £1000, if gases are to be bottled and transported, the sum of £200 would have to be added to overhead charges for interest and depreciation at 20 per cent. In plants for oxy-hydrogen cutting the cost would be lower in labour, whilst low-power compressors only would be needed and no gas bottles, making a difference of £500, approximately, in capital expenditure.

The largest item is cost of current, but there are cases where this is cheap, or even surplus power.

The total in such a case might be £1500 for overhead charges. Hence cost of oxygen=40s. per 1000 cb. ft.; or if shared by hydrogen, 20s. per 1000 cb. ft. for oxygen, 10s. per 1000 cb. ft. for hydrogen. At the present time very many works are using the electrolytic process for generating oxygen and hydrogen, both in Europe and America. Among the uses to which the gases are being put are metal welding and cutting, hydrogenation of oils, manufacture of incandescent filament lamps, metal brazing, oxy-acetylene welding, smelting work, steel production, synthetic ammonia, accumulator works. The various Government aircraft stations are equipped with electrolytic generators, and the uses of the gases are extending rapidly.

Although it is a great advantage to utilise both gases on the spot, this is not always done, and sometimes the hydrogen, or even the oxygen, is run to waste.

An analysis of about 70 plants, put down on one system only, shows 18 works utilising both gases for their own manufactures, and 17 producing both gases exclusively for supplying outside consumers. Twenty are using only the oxygen, and the rest generally combine the use of one gas for their own needs with the sale of the other to outside consumers; but, obviously, on account of its value, oxygen is seldom run to waste.

The plant is comparatively simple in construction, is run with but little supervision, and generates the purest gases. The purity of the gases is sometimes the all-important factor, as in the hydrogenation of oils and in oxy-hydrogen cutting.

There can be no doubt that the electrolytic production of oxygen and hydrogen has a considerable future before it.

## MEETINGS OF OTHER SOCIETIES.

### ROYAL PHOTOGRAPHIC SOCIETY.

At the annual meeting of the Society held on March 8, Dr. G. H. Rodman was elected president for a second year, Sir William Pope and Mr. W. L. F. Wastell were elected vice-presidents, and most of the members of the old council were reappointed. The report of the year's work showed an exceptionally large accession to the membership. This will afford considerable financial relief to the Society, whose expenses naturally reflect the changed conditions produced by the war; the subscription is being raised only to those members joining after 1920. The Society's Progress Medal was presented to Mr. F. F. Renwick "in recognition of his researches in the chemistry and physics of photography." Previous recipients of the medal include Abney, Hurter and Driffeld, and the Lumières.

The meeting of the Scientific and Technical Group, held on March 15, was devoted to reflex cameras. Mr. Newmsn gave a short sketch of the history of this form of camera, including a statement of the most important mechanical requirements and the usual methods of meeting them. Major A. Abrahams described the use of the camera in high-speed photography, especially of rapidly moving objects.

### SOCIETY OF GLASS TECHNOLOGY.

During the forenoon of the meeting held in Newcastle-on-Tyne on March 16, a visit was paid to the works of Messrs. Lemington Glassworks, Ltd., Lemington-on-Tyne, to see the manufacture of electric-lamp bulbs and glass-tubing. Of especial interest were the Westlake automatic machines, which turn out bulbs at the rate of 1000 per hour.

The first paper at the afternoon meeting was "Note on the Corrosion of Fireclay Refractory Materials by Glass-making Materials," by Mr. D. Turner and Prof. W. E. S. Turner. The authors found that even at 800° C. soda ash attacked fully-burnt fireclay, and that particles of a certain size lost 11 to 18 per cent. of their weight within three hours. Salt cake was not nearly so active as soda ash, as it took up only 1 per cent. of the fireclay at 800° C. The results indicated a direct correlation between the corrosive action of alkali salts and their temperature of decomposition, those decomposing at the lowest temperatures, e.g., nitrates, being the most corrosive.

In a paper on "The Clouding of Glass," Dr. M. W. Travers described a research undertaken to determine the causes of the clouding of soft soda-glass during lamp-working operations at a short distance from the hottest zone. As no clouding effect was observed when pure hydrogen and air were used, he concluded that clouding was associated with some impurity in the gas, and further experiments showed that sulphur dioxide was the chief cause. The remedy lay in purifying the gas from sulphur, as was practised by the South Metropolitan Gas Co. Mr. W. W. Warren drew attention to the difficulty caused by the presence of sulphur in



the working-pot of the Westlake machine, and Mr. F. F. S. Bryson stated that the Glass Research Association had been considering the same problem.

A paper by Miss E. M. Firth and Prof. Turner on "The Shrinkage, Porosity, and Density of British Fireclays after Firing at 1500° C." was postponed owing to lack of time.

Prior to the reading of the papers the chairman, Dr. Travers, referred to the death of Lord Moulton, and it was unanimously resolved to record in the minutes the Society's appreciation of the great services he had rendered to science.

#### THE FARADAY SOCIETY.

A meeting of the Faraday Society was held in the rooms of the Chemical Society at Burlington House on March 22, when Prof. A. W. Porter delivered his presidential address on "The Scientific Work of the late Lord Rayleigh."

Lord Rayleigh was the first vice-president of the Faraday Society, and but for ill-health he would have been its president. His interests in physics were very wide, and the recently published collection of his researches contains more than four hundred papers. He was equally distinguished as a theorist and as an experimentalist, and though some of his experimental work necessitated most elaborate apparatus requiring consummate skill for its design and use, he always took the keenest delight in the design of the simplest apparatus with which most valuable results could be obtained. Considering some of Lord Rayleigh's elaborate experimental work, Prof. Porter pointed out that, although at the beginning of his work the absolute value of the ohm was only known to within 4 per cent., his classical determination gave a value differing by only 1 part in 10,000 from the most recently determined figure. Similarly, he determined with certainty the fourth figure of the electrochemical equivalent of silver, although the previous work had left an uncertainty of 2 per cent. Another example of this type of work was the famous discovery of argon.

Turning to the simpler experimental methods, Prof. Porter mentioned the simplification introduced into interferometry by abolishing the necessity for an accurate parallel movement between two plates. Another interesting observation was that the fouled surface of a liquid may be cleaned sufficiently for conducting surface-tension measurements by the simple expedient of scraping it with a piece of paper or with an expanding ring.

Referring to Lord Rayleigh's interest, from 1871 onwards, in the problem of the colour of the sky, the speaker foreshadowed useful developments of his work which he hoped would soon be published by Captain Paris. Lord Rayleigh also explained the cause of the gorgeous colours often to be seen in crystals of potassium chlorate, and the arguments he adduced are applicable to the reflection of X-rays by crystals.

In his work on capillarity and the intrinsic pressure in liquids, Lord Rayleigh did not consider very deeply the theoretical aspects of the phenomena or make much advance on the ideas of Laplace. Prof. Porter then showed that if the assumption be made that the force of attraction between two atoms, whose centres are at a distance of  $r$  from each other, varies inversely as  $r^n$ , and if it be assumed that the value of  $n$  can never become less than a fixed quantity  $\sigma$ , then measurements both of surface tensions and of intrinsic pressures show that  $n$  must be about 8.3. It was, however, necessary to point out that it is improbable that the centre of the atom can be regarded as the centre of the attraction.

Lord Rayleigh was always keenly interested in hydrodynamics and made extensive use of the method of dimensions, a particular case of which is

known as "the principle of dynamical similarity." When this is applied to the sand-blasting of glass, it is readily seen that, on the assumption that rupture occurs when a particular stress is exceeded, independently of its duration, the rupture of the glass depends on the velocity of a particle of sand, and not on its size.

Mr. S. Field read a comprehensive paper on "The Electrolytic Recovery of Zinc," in which he described the state of the zinc industry both at home and abroad, and discussed the relative merits of the electrolytic and distillation processes. A detailed account of the electrolytic process followed, together with figures of working costs. The author believes that the employment of electrolytic extraction, by rendering possible the use of low-grade ores, might ameliorate the present depressed state of the industry in Great Britain and increase the national wealth. Mr. W. E. Hughes presented the first part of a paper on "The Forms of Electro-deposited Iron and the Effect of Acid upon its Structure," which dealt only with deposits from a bath composed of ferrous and calcium chlorides. The author showed that the structures found in iron deposits formed in such a bath are varieties of two general and distinct types—the normal and the fibrous—the latter being characteristic of deposits formed in acid or in agitated solutions. It was further shown that macroscopic features correspond with a macroscopic structure, a fact which may be used to facilitate control of the process. The last paper, by Prof. Alex. Findlay and Mr. V. H. Williams, was entitled "Notes on the Electrolytic Reduction of Glucose." Negative results were obtained in an attempt to obtain by electrolysis the hexahydric alcohol corresponding with glucose; both with graphite and with lead electrodes, glucose is reduced to formic acid and a pentose.

#### SOCIETY OF PUBLIC ANALYSTS.

A meeting was held in the Chemical Society's Rooms on April 6, Mr. A. Smetham presiding.

In a paper on the "Estimation of Strychnine in Scale Preparations containing Quinine and other Cinchona Alkaloids," Messrs. T. F. Harvey and S. Back discussed existing methods for separating strychnine and quinine which are only of limited value. The method recommended by the authors is based on the tartrate method of Harrison and Gair and on the ferrocyanide method of Simmonds. The greater part of the quinine is directly precipitated as tartrate from an aqueous solution of the scales, the strychnine and residual quinine are extracted from an aliquot part of the filtrate, and the strychnine is then precipitated as ferrocyanide. Characteristic residues are usually obtained. With the exception of quinine, the behaviour of which was described, the presence of small quantities of other cinchona alkaloids is not detrimental.

Mr. J. L. Lizuis described "A Method for the Determination of the Acidity of Coloured Solutions," which consists in approximately neutralising the solution, filtering through a paper previously treated with the indicator solution, adding small quantities of standard alkali or acid, and re-filtering, the procedure being continued until the filter paper gives the permanent coloration of the indicator.

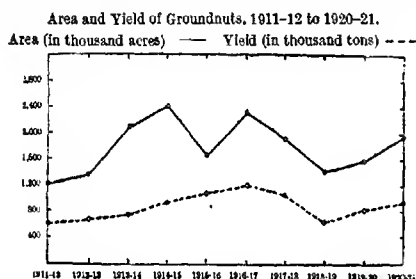
"The Action of Water on Lead" was dealt with by Dr. J. C. Thresh, who referred to the conflicting views held on this action. He has found that water free from dissolved oxygen has no action on lead, but if free oxygen be present, action begins forthwith and continues until all the oxygen is used up, the rate of action depending on the amount of oxygen present. Theoretical explanations of the reactions involved were given.

A paper on "A Colour Reaction for Aconite" was read by Dr. S. Mellaneh, who stated that the colour reactions at present known for aconite were really those of benzoic acid, and that colour reactions generally were seldom useful unless applied to pure alkaloids. The author has devised a very delicate test for aconite, involving the use of potassium ferrocyanide and formic acid. The method is apparently specific, but confirmation is being sought; it is applicable not only to the pure alkaloid, but also to the powdered root, and the author recommends it for medico-legal work, particularly in India.

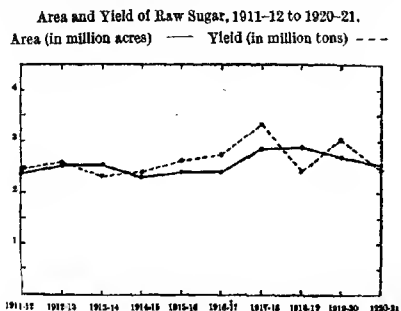
## NEWS AND NOTES.

### BRITISH INDIA.

**The Groundnut Crop of 1920-21.**—The final general memorandum on the groundnut crop of the season 1920-21 states that the total yield is estimated at 931,000 tons of nuts in shell, as against 822,000 t. last year, or an increase of 13 per cent. The total area returned is 1,951,000 acres, as compared with 1,586,000 in 1920, or an increase of 23 per cent. Of the total area under this crop in British India, 75 per cent. is in Madras, 13·5 per cent. in Burma and 10·5 per cent. in Bombay.



**The Sugar-Cane Crop of 1920-21.**—The area sown to sugar-cane is estimated at 2,553,000 acres, or 5 per cent. less than a year ago, when it was 2,686,000 acres. The estimated yield of raw sugar (*gur*) is 2,465,000 tons, compared with 3,036,000 t. last year, or a decrease of 19 per cent. Owing to deficient rainfall in certain of the producing provinces, the season has not been favourable, on the whole, for the crop sowing. The chief contributors to the total yield are:—United Provinces 52·4 per cent., Punjab 16·5 per cent., Bihar and Orissa 10·5 per cent., Bengal 8·7 per cent. and Madras 4·2 per cent. The appended chart illustrates the estimates of area and yield, as compared with the final figures for the preceding nine years.



—(Indian Tr. J., Feb. 25, 1921.)

**Cinchona and other Medicinal Plants.**—The vital need of possessing within the Empire essential raw materials, such as quinine, was strikingly brought home to us during the war. In 1914 Germany, one of the most important manufacturing and distributing countries, had full stocks in hand, with facilities for securing (during the early stages of the war) enough of the raw product to meet the wants of herself and her partner-in-arms. On the other hand, the supplies of the Allies were none too plentiful, and from the first they were dependent on the goodwill of Holland, which had, and still has, the virtual monopoly in her Java cinchona plantations. The Dutch, however, needed coal, machinery, and rice from Burma, so the result was a collective bargaining by the Allies and the signing of an agreement for the supply and distribution of quinine by the Dutch Quinine Combine and the Association of Quinine Manufacturers in Allied countries. This agreement was faithfully kept—although at some pecuniary sacrifice to the Dutch—until the end of the war. After the armistice the short supply of quinine led to hectic speculation by holders in second and third hands, and Government control became impossible. After decontrol prices became more stable, and at the present time the British Quinine Corporation, working in conjunction with the Dutch Combine, practically controls the British quinine market. Although prices have declined as a result of decontrol, the production of bark and quinine as a lucrative commercial proposition is apparently more firmly established than ever before. There is, therefore, a growing interest in cinchona cultivation in America, Japan, and within the British Empire.

An important scheme for developing the production of quinine in India was initiated during the past year, of which an account is given in the report on the Government Cinchona Plantations and Factory in Bengal for 1919-20, published in the *Calcutta Gazette* of December 8, 1920. According to this report it is probable that Burma will in time become an important source of the drug for India or the Empire. Owing to the strain of the war period, cinchona crops in Bengal have been much reduced. In 1919-20 the yield was 513,043 lb., and a still smaller harvest is anticipated for 1920-21; but this is all to the good of the plantations. The total area under cinchona is 3080 acres, of which 2319 acres is comparatively young. There are approximately 3,610,000 cinchona trees on Munsong estate and 1,480,000 on Mungpoo, making a total of 5,090,000. During the year under review 149,835 lb. of Mungpoo bark and 334,410 lb. of Munsong bark were worked up, besides 514,540 lb. of bark obtained from Java for the Government of India. These showed on analysis a total of 20,588 lb. quinine sulphate on the Bengal grown bark and 31,571 lb. on the imported bark. The products actually obtained were 47,724·25 lb. of quinine sulphate, 1022 lb. of hydrochloride (equivalent to 1160 lb. of sulphate), and 15,166 lb. of cinchona febrifuge (containing quinine equivalent to 2226 lb. of sulphate), making in all an average yield of 98 per cent. on analytical results. The total cost of the total harvest was Rs. 97,497, but the actual market value was Rs. 2,74,670; and it is pointed out that the purchase of the same quantity of bark in the market would have cost the Government Rs. 1,77,174 (*plus* freightage and carriage charges) more than it actually did.

Ipecacuanha, digitalis, and belladonna are being grown experimentally in Burma. Of the first-named, 76,000 plants are being cultivated, and during the past year the older plants seeded well and all the seed has been sown. It is still uncertain if the cultivation of ipecacuanha can be made to pay. The digitalis crops at Mungpoo did very well until heavy hail storms destroyed 50 per cent. of the leaf. To obviate this havoc in future, a portion

of the 1920-21 crop will be planted in partially-cleared forest only. European methods of drying the leaf have been employed successfully, and the product is of high quality. Moreover, with digitalis at its present price the requirements of India could easily be met by home-grown supplies. The cultivation of belladonna is only in its infancy, and good results are expected, but chenopodium culture has been discontinued owing to the low yield of oil. The collection of lycopodium from the natural supply on Mungpoo has also ceased as there is apparently no demand.

**Department of Industries.**—A Board of Industries and Munitions was constituted in February, 1920, as a temporary organisation to close the war commitments of the Indian Munitions Board, to take over some of the work of the Commerce and Industries Department and the Public Works Department, and to start the organisation of the new Department of Industries. This it has done, and consequently the board was merged into the new Department by a resolution of the Government of India dated February 15, 1921. The new department is under the charge of the Hon. Sir Thomas Holland, who is a member of the Governor-General's Executive Council.

**The Development of the Indigo Industry in Assam in conjunction with Tea and other Crops** is the title of Indigo Publication No. 8, of the Agricultural Research Institute, Pusa, written by Mr. W. A. Davis, Indigo Research Chemist to the Government of India.

Java indigo grows more luxuriantly in Assam than in Bihar, the yield of unusually high-quality green plant and seed per acre being extraordinarily high. Bihar has been almost entirely dependent on the United Provinces and Assam for its supply of indigo seed since the war. A small indigo factory was installed on the estate of the Panchnoi Tea Co. in 1919, and the yield of plant per acre, the leaf percentage on the weight of the plant, and the indigotin content have proved unusually high. In Bihar the bacterial-steeping conditions are usually good and give rise to a 70 per cent. efficiency of extraction, but in Assam the yield and quality of the indigo at first obtained were so poor, owing to the difficulties caused by green vats and bad settling, that the enterprise appeared likely to prove a failure. These difficulties have been traced to the fact that the water used for steeping at Panchnoi was obtained from a small river rising in the hills only a few miles away. The bacterial character of this water changed very rapidly with variations of rainfall in the hills; the water was very soft and, as a rule, was extraordinarily deficient in the indican-hydrolysing types of bacteria which are essential for good steeping results. An increase in the duration of steeping at Panchnoi gave much better results, the difficulty of green vats and bad settling disappeared entirely, and a very high yield of greatly improved quality was obtained. It is of the greatest importance to ascertain at each factory in Assam the duration of steeping each day which gives rise to the maximum produce and quality, and a simple colorimetric test has been devised for this purpose which permits the production in Assam of high-quality indigo, well up to the Bihar standard.

The profits to be derived from growing indigo in Assam are considerable, and with favourable working the product could not only compete with synthetic indigo, but displace it completely from the market. In addition the great value of the seed, or refuse from the steeping vats, as a rapid-acting nitrogenous manure for promoting the leaf-growth of other crops must be taken into account. The growth of indigo in Bihar is so poor that the seed obtained from 4-5 acres of indigo land only suffices to manure 1 acre of land, whereas in Assam, owing

to the luxuriant growth of indigo, the seed from 1 acre would be sufficient to manure 1 acre of land. Indigo could be grown with great advantage on newly-opened or newly-cleared tea-land, and would enable considerable profits to be made in the preliminary 4-5 years before the tea bears. Seed has also proved an admirable manure for sugar-cane, the growth of which in Assam is likely to develop considerably in the future, and indigo could be grown with particular advantage on newly-opened sugar estates. The growth of flax in conjunction with indigo in Assam is also worthy of consideration, for the flax could be grown and manufactured in the cold weather, the indigo vats would be suitable for retting, and this crop would utilise labour advantageously in the off-season. In China and Japan there is an immediate and considerable market waiting to be supplied with indigo in the proper form, i.e., a stable 20 per cent. paste such as can now be prepared readily. It is of the greatest importance that the output of natural indigo from India should be increased as rapidly as possible, for the present annual output from Bihar is less than 5 per cent. of the requirements of China and Japan.

#### FRANCE.

**Organisation of Chemical Societies.**—At the last general meeting of the Société de Chimie Industrielle, M. Paul Kestner, president, reviewed the work of the Society, put forward certain proposed modifications in its constitution, and explained its relationship to other French chemical organisations. The modifications include the formation of groups or sections corresponding to the various branches of applied chemistry; each group will appoint a president, vice-president, and secretary to hold office for three years, who will report monthly to a full meeting of all members of the group; and there will be a general meeting of all the groups once a year. The proposal to publish a joint abstract journal for pure and applied chemistry has been abandoned, and it has been agreed that the State subsidy granted for this purpose shall be shared by the *Bulletin de la Société Chimique* and *Chimie et Industrie*, which will publish abstracts in pure and applied chemistry, respectively.

The three chief chemical organisations in France are:—(1) Le Comité National de Chimie, which co-ordinates research work in pure and applied chemistry with a view to assisting Le Comité National des Recherches. (2) La Fédération Nationale des Associations de Chimie, founded in 1919 to co-ordinate the scientific, technical and economic activities of the following seven societies: La Société Chimique de France, La Société de Chimie Industrielle, L'Association des Chimistes de Sucrierie et de Distillerie, La Société des Experts Chimistes, La Société de Chimie physique, La Société de Chimie biologique, and L'Association des Chimistes de l'Industrie textile. (3) L'Union Internationale de la Chimie Pure et Appliquée, also founded in 1919, and L'Office Internationale de Chimie, the functions of which were detailed in our issue of July 31, 1920 (p. 251 B).

**Industrial Notes.**—*Chemical Industry.*—Except for a more active inquiry for superphosphate, market conditions remain practically unchanged. The factories have reduced their production by 40-50 per cent. The Lyons Fair was again a great success, and it is reported that most probably Lyons will become the centre of all fairs in France, and thus take the place of Paris, where in future a yearly exhibition of artistic products will be held. Lyons will be to France what Leipzig is to Germany.

*Metallurgy.*—The production of cast iron in France during 1920 was 3,317,371 metric tons, of

which 1,355,000 t. came from the recovered province of Lorraine; this compares with 5,311,316 t. in 1913 (exclusive of Lorraine). Of steel, 2,961,508 t. was produced, including 1,098,000 t. from Lorraine, against 4,635,166 t. in 1913. Although inquiries are more numerous the metallurgical market shows no decided improvement, and prices remain uncertain as no decision has yet been reached concerning the price of metallurgical coke.

**Cotton Cultivation.**—The cultivation of cotton is progressing very well in the French colonies; Algeria, in particular, is making good headway and will soon have about 2700 acres under cultivation. Great efforts are being made in Désirade Island, near Guadeloupe, as well as in French West Africa, in Togo, and in Cilicia.

**The Window Glass Industry.**—Owing mainly to the German invasion of Northern France the French production of window glass fell 75 to 80 per cent. during the war. Consequently imports of window glass, largely from the United States, increased very greatly, viz., from 3640 metric tons in 1913 to 8142 in 1915, 8612 in 1917, 47,900 in 1919, and 34,169 in the first nine months of 1920. The exports fell from 6116 t. in 1913 to 830 t. in 1915, and 525 in 1919, but increased to 3800 t. in the first nine months of 1920. During the war two new window-glass factories were established in the interior of France—the Société des Verreries Mesmer, at Andrezieux (Loire), and the Société des Verreries du Centre, at Montluçon (Allier). Practically all the factories in the devastated regions have been reconstructed, and the French production of window glass is now much in excess of home requirements. Exporters find it difficult to make firm quotations owing to unstable coal prices and unsettled labour conditions. In December last several factories in the north had to close down owing to lack of orders and dear coal. Large stocks are accumulating, and the Government is being urged to fix a special low price for coal used by glass manufacturers, in order to assist them in competing with their Belgian rivals.—(*U.S. Com. Rep.*, Jan. 26, 1921.)

#### CANADA.

**Maple Sugar, Honey, and Wax in Quebec.**—The production of maple sugar in Quebec in 1920 amounted to 13,393 long tons, of an estimated value of \$7,000,000. Apiculture is also a flourishing industry, and the output of honey and wax was 1313 t. and 12 t., respectively.—(*Official.*)

**Mining Developments in Vancouver Island.**—The Canadian Pacific Railway Co. is about to undertake a survey of two million acres of mineral-bearing land owned by it in Vancouver Island, the work to be carried out under the direction of the Trail Smelter Co., also owned by the C.P.R. It is considered probable that the survey may lead to the erection of a smelter on Vancouver Island. Particular attention will be given to the development of the coal deposits.—(*Official.*)

**Proposed Asbestos Control in Quebec.**—The Quebec Government is reported to be considering the question of controlling the asbestos industry of the province in the same way as the pulp-wood industry, i.e., by attaching to mining leases a condition that the mineral won shall be worked up, either wholly or partially, in factories erected in the province. Quebec provides 85 per cent. of the world's supply of asbestos. In 1919-20, 174,521 tons, valued at \$11,758,234, was exported; but very little is manufactured in the province.—(*Official.*)

**Coal Production in 1920.**—The Canadian Bureau of Statistics reports that the output of coal in 1920 was 16,968,568 short tons, the highest production ever attained; in 1919 it was 13,919,096 t. Alberta produced 41 per cent. of the output, Nova Scotia 37.8, British Columbia 18.3, Saskatchewan 1.9, and

New Brunswick 1 per cent. By kinds of coal, the increases in output amounted to 0.3 per cent. for anthracite, 16.0 per cent. for bituminous, and 5.6 per cent. for lignite. In December, 1920, 22,848 underground and 8508 surface workers were employed at the mines. Exports increased to 2,558,223 t., compared with 2,070,050 t. in 1919, Nova Scotia being the chief coal-exporting province. Imports from the United States rose from 16,982,773 to 20,815,596 t., Ontario being the largest importer. The total coal supply amounted to 35,225,941 tons.

#### SOUTH AFRICA.

**Output of Base Minerals in 1920.**—Returns prepared by the Government Mining Engineer give the output of base minerals in South Africa for 1920 as follows:—

	Output, Tons.	Value, £
Arsenic, white..	11	655
Iron ore ..	2,564	811
Lead ore ..	587	5,787
Manganese ore ..	67	277
Zinc ore and concentrates ..	2,349	15,614
Asbestos ..	7,112	114,195
Granum ..	260	1,446
Graphite ..	73	2,339
Iron pyrites ..	3,469	5,014
Magnesite ..	1,418	3,780
Mica ..	0.77	500
Ochres, etc. ..	536	1,040
Talc ..	682	2,227

The total value of the output was £153,694.—(*S. Afr. J. Ind.*, Feb., 1921.)

#### AUSTRALIA.

##### Queensland.

**State Enterprises.**—The Government of Queensland has undertaken a considerable number of enterprises, under the direction of a Minister for State Industries, which include, among others, pastoral stations, sawmills, coal mines, iron and steel works, canneries, and a produce agency. Last year's balance-sheets for the whole of the enterprises showed assets valued at £1,976,090 and liabilities £1,743,124. The State pastoral stations cover almost 30,728 sq. miles of territory; the coal mines and the steel works are yet in the development stage; and much attention is being given to the development of the cannery industry, which will utilise fruits from the new settlements for returned soldiers formed by the Repatriation Department. The State Forestry Department is also making steady progress, and the director is urging the development of the Forest Products Laboratory. Among the products of the Queensland forests are:—Eucalyptus kinos containing 70 per cent. tannic acid; Cupanias containing commercial saponins; the Bauhinias yield gum arabic; the citron-scented Ti trees produce important essential oils; the Alstonias yield a caoutchouc; the Macrozamia a useful starch and a vegetable wool; and the Grass tree a valuable resin. In addition, there are millions of acres of apparently useless wood which could be distilled for wood spirit, acetic acid, etc.

**The Prickly Pear.**—This terrible pest is spreading very rapidly over large districts in Queensland, and so far no satisfactory means of eradicating it has been evolved. The following are some of the methods which have been tried experimentally:—(1) The insect *Dactylopius tomentosus* will under certain circumstances attack the pear and destroy it, but quantities of the insects sufficient for large-scale work have not been obtained. (2) Felling the trees by cheap indentured labour. (3) When properly applied, arsenious chloride gas kills the tree completely, but it is difficult to prevent it rising too quickly. (4) Applying the sun's heat by means of powerful lenses. This method has been tried in California but it is expensive. (5) Sub-

mersion under water for three weeks is quite effective. It has been proposed to dam certain rivers, flood the districts for a time, and to use the water afterwards for irrigation purposes. (6) The Director of Forests proposes to master the growth of prickly pear by means of an overgrowth of drought-resisting, umbrageous, leaf-littering trees such as the southern silky oak, camphor laurel, and Canary Island or other pines (*cf.* J., 1919, 455 B).

#### UNITED STATES.

**Vitreous Enamelling.**—A valuable bibliography on the vitreous enamelling of iron and steel has been prepared by Dr. C. J. West and printed in the *Journal of the American Ceramic Society*. As the previous list, by J. C. Branner, covered the literature up to 1906, the present one begins with the year 1907.

**Visit of Madame Curie.**—Madame Curie is about to visit America, and it is proposed by certain women's organisations to present her with one gram of radium, estimated to cost \$100,000. Although Madame Curie played a leading part in the discovery of this element, she does not possess any of it to-day, and the object of the gift is to enable her to continue her researches.

**Food Research Institute.**—A food research institute is to be established at Stanford University, California, by the Carnegie Corporation of New York. Dr. C. L. Alsberg, chief of the Bureau of Chemistry of the United States Department of Agriculture, has been appointed director, and the Corporation will provide \$700,000 for the support of the Institute for 10 years. Work will be started on July 1. The University laboratories will be utilised, and duplication of any other work in the field of nutrition will be avoided.

#### GENERAL.

"Antimony 1913-1919," issued by the Imperial Mineral Resources Bureau as a 34-page pamphlet, describes the chief antimony minerals and their occurrence within the British Empire and in foreign countries, and gives such statistics of production as are available. Antimony is chiefly used in the manufacture of alloys and except in times of war, when large quantities of shrapnel bullets containing about 12 per cent. are required, there does not appear to be a greatly increasing demand. Of the compounds, the tetroxide is used in the ceramic industry for making opaque white enamel, the trioxide (known as antimony white) is used as a pigment, whilst other pigments are antimony vermillion (a red trisulphide of antimony), antimony yellow, produced by the slow oxidation of the sulphide, and antimony blue, resulting from admixture of the above with other minerals. Antimony sulphide is used extensively in the manufacture of rubber goods.

The chief antimony-producing countries in order of importance are China, France, Mexico, Italy, and Australia, but China has long since predominated, and during the war the output of crude regulus and refined antimony greatly increased—from about 13,000 tons in 1913 to about 35,000 tons in 1917. A considerable proportion of the world's output of refined antimony is produced in England from imported raw material. Its high quality is recognised throughout the world.

**National Association of Industrial Chemists.**—The report of the General Secretary presented at the fourth annual meeting held at Sheffield, Mr. A. B. Searle presiding, states that the Association has successfully come through the recent very critical period caused by industrial depression, and that it now possesses nearly 1100 members. Owing to the need for economy, it has been decided to post-

pone the publication of a magazine. Members have taken full advantage of the Advice Bureau; a special committee was appointed early last year to draw up a scale of minimum salaries, and as a result of assistance given by the Association over one hundred members who were receiving less than the adopted minimum succeeded in obtaining increases in salary. Another committee was formed to discuss preliminaries with the British Association of Chemists with a view to amalgamation, and negotiations are still proceeding. It is held to be essential to the interests of all British industrial chemists that a single, strong, fully representative organisation should be formed. The Employment Bureau has assisted members in obtaining situations, and only nine are now out of work.

**Output of Iron and Steel in the United Kingdom in 1920.**—The output of pig iron in 1920 was 8,000,700 tons, an amount which, though 8 per cent. more than the 1919 output—7,398,000 t.—is less than that of any war year and over 20 per cent. less than in 1913. A total of 9,055,600 t. of steel ingots and castings was produced, compared with 7,894,000 t. in the previous year. Production in both cases would have been considerably higher but for the coal strike, which reduced the monthly average during the last quarter of the year. In 1913, a record year, the production of pig iron and steel was 10,260,000 and 7,663,900 t., respectively, and the present proportions of the output indicate that the steel capacity is far in excess of the pig-iron production. The output of metallurgical coke in 1920 was 12,494,400 t., or 1,000,000 t. more than in 1919.—(*Iron and Coal Tr. Rev.*, Jan. 7, 14, Mar. 25, 1921.)

**The Austrian Cement Industry.**—The output of the Austrian cement industry for 1920 is estimated at 120,000 to 130,000 tons. Owing to more plentiful supplies of fuel, the production has been increased by 27 per cent., but it is still far below the capacity of the industry and supplies only about 22 per cent. of the normal consumption.—(*U.S. Com. Rep.*, Jan. 20, 1921.)

**The German Cement Industry.**—Government operation of the German cement industry was provisionally abolished in July, 1920, owing to the pressing demand for cement having been satisfied, but the Government still fixes prices for the domestic market. The price of cement increased from 35 to 333 marks per metric ton between August, 1914, and August, 1920, and in December, 1920, it was fixed at 310-333 mk., according to district, lower prices being fixed for cement supplied on Government contracts. During the middle of 1920 cement production increased, partly owing to the resumption of operations of the Offenbach Portland cement works, after a stoppage of over five years. Shortage of coal has affected the cement industry less than other industries, as it has been able to use much low-grade coal. Export of cement has been resumed.—(*U.S. Com. Rep.*, Mar. 10, 1921.)

**The Mineral Colour Industry in Spain.**—A good grade of white lead has been manufactured for many years by the Real Cia. Asturiana de Minas, of Barcelona, a company which owns several large works and can supply most of the Spanish requirements. A small amount of good white lead is also made by the Vincente Fité y Cia at Mataró. Lithopone is made in large quantities principally by the Industrias Químicas Alhinana Argemí company, of Barcelona, which produces a very fine product. The prices of white lead and lithopone are fixed by the Spanish firms so that they are always somewhat lower than those of the imported products. Blanc fixe is made by a number of small firms: the firm of M. and G. Forst, of Barcelona, produces it as a by-product in the manufacture of

hydrogen peroxide. As the blanc fixe made in Spain is not of the best quality, the paper factories import it from England, France, and Germany. Chrome yellow of various shades is made in a large number of small works which also produce chrome green by blending the yellow with imported Prussian blue. Chrome yellow, zinc white, zinc yellow, zinc green, etc., are made at Barcelona by the Fabricacion General Espanola de Colores, Gerardo Collardin; red lead and litharge are made in large quantities and at competitive prices by the Real Cia. Asturiana de Minas, the Vicente Fitó y Cia., and by a few other small firms in Catalonia.—(*Z. angew. Chem.*, Feb. 4, 1921.)

**The Swiss Dye Industry in 1920.**—Large quantities of dyes and intermediates were exported from Switzerland during the early part of the year and the manufacture of intermediates not hitherto made proved to be very profitable. The high value of Swiss currency, however, hindered trade with certain countries and made it impossible with others. Nevertheless the situation remained satisfactory until the autumn, when the crisis in the textile industry led to a reduced consumption of dyes; by the end of the year the stagnation had become general. The labour situation was good and there were no strikes for increased pay; wages, however, were raised twice during the year. In gauging the future of this industry, it must be remembered that wages are many times greater than those paid in competing countries, and that coal is many times dearer. In view of the contingencies that foreign import duties may be raised, or that economic conditions in Switzerland may affect the export trade, the controlling dye interests in Basle have purchased a factory in the United States at Cincinnati, in addition to the works acquired in England in 1919.—(*Z. angew. Chem.*, Feb. 18, 1921.)

**Coquito Nuts in Mexico.**—The coquito palm grows abundantly in Mexico in the States of Colima, Jalisco, Nayarit, and Sinaloa, where about 5000 tons of nuts are annually harvested. The oil expressed from the nuts is highly prized by the soap manufacturers of Guadalajara and Mexico, who consume the entire production. Ten years ago when the value of the nuts was little appreciated, they were sold at about \$40 per ton, but to-day they fetch \$150–250.—(*U.S. Com. Rep.*, Feb. 5, 1921.)

**Alcohol Production in the Dutch East Indies.**—The export of alcohol from the Dutch East Indies in 1919 amounted to 3,397,680 galls., and in the first half of 1920 to 1,827,100 galls., which compares with 2,685,980 galls. in the corresponding period of 1919. The advance in the trade, which is rapidly assuming greater importance, was mainly due to increased exports to Italy and France, and to a very large demand from Singapore, Hongkong, India, and Ceylon.—(*U.S. Com. Rep.*, Jan. 19, 1921.)

**Cultivation of the Oil Palm in the Dutch East Indies.**—Of late years an increasing interest has been taken in the cultivation of the oil palm (*Elais Guineensis*) in Dutch East India. Throughout the cultivated area of the east coast of Sumatra considerable tracts of land have been planted with this tree, and in 1919 existing estates were extended and new ones opened. At the end of that year 42 estates, covering an area of 13,500 acres, were devoted exclusively to this particular palm, and the total production of palm oil amounted to 830 metric tons, as against 330 tons in 1918. It is expected that during the next few years the area under cultivation will be considerably extended, and if the planting schemes now in hand are fully realised, the cultivated area should eventually yield a production of about 100,000 tons of palm oil.—(*Dutch East Indian Archipelago*, Jan. 25, 1921.)

## PERSONALIA.

Prof. L. J. Henderson has been appointed Harvard exchange professor in biological chemistry to France, and will lecture at the Sorbonne during the second half of the present academic year.

Recent appointments in the University of New York include those of Prof. H. R. Moody as professor of chemical engineering, and Prof. W. L. Prager as ordinary professor in the same faculty.

Mr. H. Salt, late of Leeds University, and now of the Forest Products Laboratory of Western Australia, is making a survey of the resources of tanning materials in that State on behalf of the local Government.

Dr. R. F. Ruttan, head of the chemistry department at McGill University, has succeeded Dr. D. G. MacCullum as administrative chairman of the Advisory Council for Scientific and Industrial Research in Canada.

Prof. K. Hess has accepted the post of director of the organic-chemistry section of the Kaiser Wilhelm Institute for Chemistry in Berlin, in succession to Prof. E. Beckman.

Prof. Pietro Corbetta, formerly professor of inorganic chemistry at the Milan Polytechnic, has died in his 72nd year.

The deaths of the following members of the Society in the United States have recently been announced:—Mr. J. Hasslacher, president of the Roessler-Hasslacher Chemical Co.; Mr. J. D. Pennock, general manager of the Solvay Process Co., Syracuse, N.Y.; and Dr. E. J. Lederle, a well-known chemist and bacteriologist.

## CORRESPONDENCE.

### THE ORIGIN OF THE SOCIETY OF CHEMICAL INDUSTRY.

SIR,—Mr. Douglas Herman, in his letter to the *Journal* of January 31 last, states that the chairman of the meeting held at Widnes at which members of the Faraday Club attended was Mr. James Muspratt, brother of Dr. E. K. Muspratt. Dr. E. K. Muspratt's brother was Dr. James Sheridan Muspratt. Mr. James Muspratt, chairman, would be Mr. James Liebig Muspratt, Dr. E. K. Muspratt's nephew, and son of Mr. Richard Muspratt, of Flint, N. Wales.—I am, Sir, etc.,

St. Albans,

MARTIN TAYLOR.

March 22, 1921.

SIR,—The letter of Mr. John Hargreaves in the *Journal* for March 15 (p. 86 R) appears on the whole to confirm my own impressions, which were written mostly from memory. The only point at issue between us appears to be the relative influence upon the formation of the Society exercised by the members of the Faraday Club and by the Widnes chemists who wished to name the new society "The South Lancashire Chemical Society"; whereas I believe that the former were mainly responsible, Mr. Hargreaves gives his vote in favour of the latter. Can we not agree to admit that the credit is due to both?

Among the men who assisted notably in the formation of the Society of Chemical Industry I certainly ought to have mentioned the late Dr. Ludwig Mond.—I am, Sir, etc.,

Lyons,

DOUGLAS DE L. HERMAN.

March 28, 1921.



## PARLIAMENTARY NEWS.

### *Safeguarding of Industries.*

A White Paper (Cmd. 1219) has been issued which sets out the proposed Ways and Means Resolutions to be discussed in the House of Commons prior to the introduction of the projected Bill dealing with "key" industries, etc.

Resolution I proposes the imposition for a period of five years of an import duty of 33½ per cent. *ad valorem* on certain articles which include *inter alia*:—(a) Optical glass and optical elements, microscopes, spectrosopes, and other optical instruments. (b) Beakers, flasks, etc. and other scientific glassware and lamp-blown ware, evaporating dishes, crucibles, combustion boats, and other laboratory porcelain. (c) Scientific instruments such as balances, pyrometers, barometers, etc. (d) Wireless valves and similar rectifiers, and vacuum tubes. (e) Ignition magnetos and permanent magnets. (f) Arc-lamp carbons. (h) Metallic tungsten, ferro-tungsten and manufactured products of metallic tungsten and compounds (not including ores or minerals) of thorium, cerium and other rare-earth metals. (i) All synthetic organic chemicals (other than synthetic organic dyestuffs, colours and colouring matters imported for use as such, and organic intermediate products imported for their manufacture), analytical reagents, all other fine chemicals, and chemicals manufactured by fermentation processes. Articles falling under any of the above descriptions which may be contained in any list issued by the Board of Trade are also included.

Resolution II prescribes an *ad valorem* duty of 33½ per cent. on "articles of any class or description in respect of which an Order by the Board of Trade has been made under any Act of the present Session for giving effect to this Resolution, if manufactured in whole or in part in any of the countries specified in the Order, or deemed to be so manufactured." The grounds on which such Order may be made are that the article in question is being sold or offered for sale in the United Kingdom (a) at prices below the cost of production, or (h) at prices which, owing to depreciation of the currency of the producing country, are below the prices at which similar goods can be profitably manufactured in the United Kingdom; and that by reason thereof employment in any home industry is being, or is likely to be, seriously affected. The term "cost of production" is defined.

### HOUSE OF COMMONS.

#### *Imports of Gas Mantles.*

Sir P. Lloyd-Greame, replying to Lieut.-Col. Sir J. Norton-Griffiths, said that 12,668 gross of incandescent gas mantles had been imported from Germany during the period January 1 to March 31, 1921. The general condition of the gas-mantle industry had been considered, and its difficulties were known.—(Apr. 4.)

#### *The Coal Crisis.*

In moving a resolution under the Emergency Powers Act, 1920, Sir R. Horne made a statement upon the coal crisis, in which he said that, following on the last coal strike, it was agreed that the owners and the Miners' Federation should attempt to find a new basis for the payment of wages, but the owners desired that any wage basis should be settled district by district, whereas the miners contended that it should be a national settlement, although that involved a pooling system under which profitable undertakings would have to subsidise unprofitable ones. After the determination of decontrol was announced the owners

gave notice that they could no longer continue on the same basis and were obliged to fix a new scale. So far the miners had refused to discuss the new rates with the owners, and this had led to the present stoppage. Apparently the issue was not one of wages, but whether there should be a subsidy or not.

Mr. Clynes claimed that the hurried determination of the decontrol of coal was the cause of the trouble, together with the magnitude and suddenness of the reductions in the rate of pay.

Mr. Lloyd George said that the Government would willingly assist in further conference, but it must be understood that there could be no maintenance of the coal industry out of taxation, no re-establishment of control, and that attempts must be made to stop the flooding of the mines.—(Apr. 5.)

### *The Paper-making Industry.*

Sir W. Seager asked if the President of the Board of Trade was aware that out of 48,000 employees in British paper mills 26,201 were totally unemployed and 16,153 on part time, owing mainly to the dumping of paper from Finland, Germany, Sweden, and Norway.

In a written answer, Sir P. Lloyd-Greame stated that he was aware of the unemployment in the industry, and although this was probably accentuated by cheap imports from the countries named, it should be noted that Swedish currency had not depreciated compared with sterling, and that Norwegian currency had depreciated relatively little.—(Apr. 7.)

## REPORT.

GENERAL REPORT ON THE INDUSTRIES AND COMMERCE OF SPAIN (DECEMBER, 1920.) By CAPT. U. DE B. CHARLES, *Commercial Secretary to H.M. Embassy, Madrid. Department of Overseas Trade. Pp. 72. London: H.M. Stationery Office. 1921. Price 1s. 6d. net.*

Spain is above all an agricultural country and produces, in addition to fruit and cereals, sugar, olive oil, and esparto grass. Both beet and sugarcane are cultivated, but the former is the more important, as it is grown over nearly the whole country; cane would be grown much more extensively were larger supplies of basic slag available from England. The sugar industry is controlled by a trust. Spain is the largest producer of olive oil in the world; nearly 4 million acres are planted with the olive tree. The most productive regions are the provinces of Jaen, Cordova, and Seville, and the oil is extracted chiefly in Malaga and Cordova. The estimated production of oil in 1919 and 1920 was 3,360,000 and 2,632,500 quintals, respectively. Not more than 25 per cent. of the production can be classed as fine-grade oil. Export of the oil was prohibited in October, 1920, in order to conserve domestic supplies. Esparto grass grows wild in the mountainous districts near Aguilas and Almeria, and about 150,000 tons is harvested each year. It is chiefly used in the manufacture of paper and rope, but experiments are being made to produce a finer fibre, and one recently obtained compares favourably with the jute yarns used in the manufacture of sacking and fine twines. Export of this grass has fallen off during the last year, but the industry could be much expanded.

The mineral resources are very great, and large areas have not yet been surveyed. In 1918 the capital employed in mining enterprises was estimated at 984,821,000 pesetas, of which 569,630,000



pesetas was held by foreigners (peseta=9½d. at par). The value of the Spanish mineral production has risen by nearly 75 per cent. during the last seven years, but the output, except that of coal, has decreased owing to industrial troubles, high working costs, and transport difficulties. Iron ore constitutes one of Spain's greatest economic reserves; the provinces of Viscaya, Santander, Almeria, and Huelva are the chief producers. The output of the province of Huelva is mainly iron pyrites. The highest-grade hematite ores are gradually being worked out, and it may soon be necessary to exploit deposits more distant from the coast. At present the industry centres round Bilbao, Santander, and Huelva, the output of pig-iron and steel is increasing, and there are now over 30 blast furnaces. The output of steel is almost entirely controlled by a combination of five manufacturing firms, called the Central Siderurgica.

The principal coalfields are situated in Asturias, Penarroya in Central Spain, and the Ebro district, the yearly production being now about 6 million tons. Much interest is being taken in the development of the coal deposits, but lack of experienced miners and modern plant has impeded progress. It is to be hoped that Great Britain will soon regain her former position as an exporter of coal, as the Spanish market is in danger of being lost to the United States. The lead and silver lead mines are mainly situated in the provinces of Cordova, Jaen, Murcia, and Badajoz, and practically the whole output of copper ore comes from the province of Huelva, where the British-owned Rio Tinto and Tharsis mines are the largest producers. Large reserves of zinc ore are stated to exist in the provinces of Santander and Murcia. Almost the whole of the manganese ore produced came from the province of Huelva, where it is found as a silicate with 28-32 per cent. manganese; the output has increased greatly owing to the cutting off of Russian supplies. The production of ores, minerals, metals, etc., in 1919 was as follows:—

	Tons.		Tons.
Coal and lignite ..	6,243,509	Zinc ores ..	103,608
Coke, etc. ..	1,027,936	Zinc metal ..	16,313
Lead and silver-lead ores ..	178,055	Mercury ores ..	24,966
Iron ores ..	5,071,249	Mercury metal ..	1,224
Iron and steel ..	835,358	Manganese ores ..	66,685
Copper ores ..	1,470,090	Superphosphates ..	135,526
Copper metal ..	34,369	Cement ..	363,450

The total value of the production at the mines was 499,662,644 pesetas, and the value of the metals etc. at the works was 566,428,923 pesetas. Other minerals found in Spain are potash (the deposits at Cuna, in Barcelona, are said to contain nearly a million tons), salt, phosphates, wolfram, tin, bauxite, ochres, graphite, sulphate of soda, tripoli, and immense quantities of marble, granite, and other building materials.

The development of Spanish industry was greatly stimulated by the war; the machinery, metal, chemical, and textile industries made the most progress, but with the return of peace this advance has not been maintained. With few exceptions, Spanish goods are still far behind the standard of imported goods, and trade is being lost by the short-sighted policy of maintaining fictitious prices.

Although labour is plentiful, the political situation, the shorter working day, and decreased output are seriously affecting trade. The reaction began in 1919, and by the end of 1920 the country was filled with alarm. The total exports in 1919 were valued at 1,316,275,000 pesetas (£52,651,000 at par); about one-half consisted of foodstuffs, and one-sixth of raw materials. The export figures for the first nine months of 1920 are about one-fifth lower than those for the corresponding period in 1919, but mineral exports exceed those of 1919 by about 50 per cent., and chemicals show a gain of 7 per cent. The value of the imports has rapidly in-

creased during the last two years, the aggregate value during January-October, 1920, exceeding those of the corresponding periods in 1919 and 1918 by 32 and 130 per cent. respectively. Imports of chemicals and fertilisers increased by 80 per cent. over those in 1919; 65,500 t. of nitrate was imported from Chile and England, and other fertilisers came mainly from the United States. The United Kingdom was the chief source of mineral colours, varnishes, and writing ink, whereas printing ink and coal-tar colours came largely from the United States. The United Kingdom also supplied practically all the tinplate, a large proportion of the imports of iron and steel, and most of the pharmaceutical products. U.K. manufacturers have a good prospect of recapturing the trade in heavy chemicals, now in Dutch and Belgian hands. British goods are preferred, and the future of British trade in Spain should be assured if local requirements are studied sufficiently.

## TRADE NOTES.

### BRITISH.

**Seychelles in 1919.**—The export of copra in 1919, 4328 tons valued at Rs.1,445,722, was considerably greater than in previous years. The cultivation of the coconut has been improved, attention has been paid to its diseases, and experiments have been made on the use as fertilisers of seaweed, ash from the distillation of essential oils, and guano. Nineteen times more rubber was exported than in 1918, the 1919 crop being 27,260 lb. Para rubber grows well, if slowly, but the tapping is badly done. Essential oils are important products, and in 1919, 44 factories produced 24,430 litres of cinnamon-leaf oil, which fetched high prices on the London market. *Ocimum viride* oil, produced experimentally, was found to contain over 50 per cent. of thymol and the cultivation of the plant has been taken up. Ambrette seeds are no longer grown, as they are produced more cheaply elsewhere. Fibre from *Pourcroya gigantea* is made in increasing quantities, and the plant, like sisal, could be grown on a large scale. The exports from the colony were valued at Rs.2,101,377, the chief destinations being France (Rs.1,113,737) and the United Kingdom (Rs.705,422). Imports were valued at Rs.1,125,316, those from the United Kingdom showing an increase of Rs.53,795 compared with 1918.—(*Col. Rep.—Ann.*, No. 1061, Feb., 1921.)

### FOREIGN.

**Proposed Japanese Duties on Soda Products.**—According to *The Times*, the Japanese Government has introduced a Bill to increase the import duty on caustic soda by 100 per cent., and that on soda ash and natural soda by 400 per cent. Such increased duties would affect the Japanese glass industry, which exports glass to the value of £1,500,000 yearly, and also British manufacturers of soda compounds.

**Soda Trade in Manchuria.**—There is a considerable demand for soda ash, bleaching soda and caustic soda in the Mukden district; Great Britain and its colonies supply all three of these, Japan only the first two, and the United States practically none. According to the customs returns, 54,286 tons of soda compounds was imported into China during 1919, of which 6316 t. was disposed of in Manchuria. The British Empire supplied 74.5 per cent. of the total Chinese imports, Japan 14 per cent., and the United States 11 per cent.—(*U.S. Com. Rep.*, Jan. 14, 1921.)

**Japanese Tanning Industry.**—This industry developed considerably during the war, and large quantities are now used of galls, oak bark, mangrove bark, quebracho wood and various extracts, principally catechu and persimmon. The persimmon extract is made from a tree grown for the purpose, and an exportable surplus is produced. Imports of tanning materials and tanning extracts in 1919 amounted to 24,876 and 7291 short tons, respectively; they were derived mainly from South Africa (57%), British India (29%) and China (10%); and the sources of the imported extracts were:—Dutch East Indies (34%), Argentina (22%), and the Straits Settlements (21%).—(*U.S. Com. Rep.*, Jan. 22, 1921.)

**State of Japanese Trade in January, 1921.**—The state of the Japanese export trade was still unfavourable in January, and there was little prospect of improvement; imports were also small, and are considered certain to exceed exports for a time. The general financial position, however, was much better than was anticipated at the end of 1920. Stocks of copper amounted to 30,000 tons and were increasing. Reductions had been made in the prices of all products of the Government iron foundry at Yawata, but as the quotations were above market level, consumption can scarcely be affected; stocks in hand at the foundry amounted to 370,000 tons. Such large stocks of European paper had accumulated that manufacturers have reduced production as far as possible, and a fall in prices was anticipated. The depression in the match industry was most severe; many of the smaller manufacturers have suspended operations; all the large companies have made losses, exports have declined by 75·6 per cent., as China, formerly a large customer, is developing an industry of its own, and Swedish competition is growing. Belgium was competing strongly in the glass trade and exporting large quantities of window glass; local prices have been reduced but the decline in export was leading to a diminished output. German reparations dyes were still arriving, but the demand was negligible and stocks were estimated at 290 long tons (aniline dyes 197 t., alizarin 18 t., and synthetic indigo 78 t.).—(*U.S. Com. Rep.*, Mar. 10, 1921.)

**Siamese Imports of Chemicals and Drugs.**—Chemicals and drugs are not manufactured in Siam, and it appears likely that increasing quantities of these materials will be imported in the near future. The imports of chemicals in recent years (the Siamese fiscal year ends on March 31) have been:—1917-18, 949·8 metric tons (£32,245); 1918-19, 1088·4 t. (£52,039); 1919-20, 916·4 t. (£42,995); whilst imports of drugs were:—1917-18, 903·8 t. (£98,910); 1918-19, 980·6 (£137,528); 1919-20, 971 t. (£124,319). The values given assume normal rates of exchange. Hitherto the bulk of the Siamese trade in chemicals and drugs has been in European hands, but the proportions supplied by the United States increased from 1·7 to 4·1 per cent. of the chemicals in 1918-19 and 1919-20, and from 0·7 to 3·5 per cent. of the drugs imported during the same period.—(*U.S. Com. Rep.*, Feb. 7, 1921.)

**Chinese Trade in Rubber Goods.**—Practically no rubber goods are manufactured in China, and the imports of crude rubber and gutta-percha are extremely small; in 1919, a record year, they were only 40 tons. The market for tyres, though limited to the few districts with good roads, is steadily developing and the value of the net import of motor-car tyres into the Shanghai district is ten times greater than in 1913. Tyres and other rubber goods rapidly deteriorate on keeping, and therefore big stocks cannot be held. There is an excellent demand for rubber goods such as gloves, hot-water bottles, vulcanite, etc., but the market for rubber

foot-wear and clothing is small, although proofed fabrics apparently sell more readily. The chief port of entry for rubber and rubber manufactures is Shanghai, followed by Dairen, and the import duty is 5 per cent. *ad valorem*, plus 5 per cent. of the duty charges for wharfage and warehouse dues.—(*U.S. Com. Rep.*, Jan. 19, 1921.)

## COMPANY NEWS.

### BRITISH DYESTUFFS CORPORATION, LTD.

The second ordinary general meeting was held on April 1, in Manchester. Sir H. Birchenough, who presided, referred to the late Lord Moulton's services to the dyo industry, and then commented upon the report and accounts. In spite of the fact that only a part of the capital had been remuneratively employed, the year ended October 31 last was a satisfactory one. The profit, after allowing for depreciation and taxation, was £552,100 (issued capital £9,202,093), out of which the full 7 per cent. dividend on the preference shares had been paid; a dividend of 8 per cent. on the preferred ordinary shares was recommended, leaving £203,304 to be carried forward, as against £58,826 brought in. The sum of £239,366, inclusive of capital expenditure, had been spent on research during the past two years, and preliminary expenses figured among the assets at £251,551. On October 31 stocks in hand were valued (at cost or market price) at £3,998,543. The sum of £593,486 had been set aside to cover depreciation during the past two years, and in view of present trade conditions the directors had decided to suspend expenditure on constructional work.

The policy of the Corporation was to establish and develop a dyestuff industry which would gradually supply all the requirements of British consumers, and that progress in this direction had been made was evidenced by the fact that the increase in output was 51 per cent. above that for the previous year. Trade was so active during the early part of the year that all the demands of the colour-users could not be met, although sufficient dyes were supplied to enable them to take advantage of that period of unexampled prosperity. As the year 1920 advanced conditions became gradually worse owing to the flood of German imports and the general decline in trade. Every effort had been made to develop the Research Department under the direction of Prof. A. G. Green and Dr. R. Robinson, but the system of dual control by two managing directors had proved unsuccessful; it had been decided to replace the latter at an early date by one individual, but in the meantime the executive powers of the board would be vested in the chairman and two of the new directors, Sir W. Alexander and Mr. Vernon Clay, chairman of the Colour Users' Association (the third new director being Field-Marshal Sir W. Robertson). The late managing directors had been appointed ordinary members of the board, and the immediate duty of the new executive committee was to organise adequately the staff of the Corporation.

On questions being invited, Mr. J. Parker Smith moved that the adoption of the report should be deferred pending the report of a committee of investigation, but that the accounts be approved and the dividend on the preferred ordinary share be paid. He criticised very adversely the supersession of the two managing directors, Sir J. Turner and Dr. H. Levinstein, who had been the mainstay of the dye-making industry in this country. After a succession of speakers had spoken in favour of the amendment and several had demanded the reason

of the summary action taken against the two managing directors, the chairman said that the board could not agree to the course proposed, and that he was not prepared to argue the matter at a general meeting. The decision had been made after deliberate consideration; it was well known that the managing directors had not co-operated harmoniously, and the interests of the Corporation demanded the termination of their contracts. On a show of hands the amendment was declared lost by a narrow majority, and another amendment—that the appointment of the new directors be not confirmed—was rejected on a poll.

**BORAX CONSOLIDATED, LTD.**—The Rt. Hon. the Earl of Chichester, in presiding at the general meeting of this company held on March 21, said that the net profits for 1920 amounted to £476,581, compared with £442,023 in the previous year. The total of the reserve funds was £827,179 (issued capital £4,494,978, inclusive of debentures). A very large amount of ore was in sight, stocks stood at £506,617 on September 30, and the sum of £664,415 was on deposit and current accounts. A Continental works had recently been purchased on favourable terms, and other developments were contemplated. There were signs of recovery from the present bad trading conditions. Dividends on the deferred ordinary shares amount to 15 per cent. for the year.

**BRITISH ALUMINIUM CO., LTD.**—Addressing the annual meeting on March 31, Mr. A. W. Tait, chairman, stated that the company had achieved a record output and record sales during the year 1920; the great demand during the early part of the year could not be fully met. The company took over the entire Government stock of aluminium, all of which had now been disposed of. The period of financial stringency, which began last August, led to a serious disturbance of the motor trade and of the aluminium industry. Adverse conditions still prevailed, although there were signs of improvement; meanwhile the company was restricting its output. A great future awaited the application of suitable aluminium alloys in the motor trade and in general engineering, in the electrical industry, and in the manufacture of domestic utensils. Additions had been made to the alumina works in Scotland, and to the rolling mills at Warrington. The company had again taken steps to acquire additional water power in Scotland, and had modified the scheme put forward in 1918, which did not obtain Parliamentary sanction, in such a way as to allay local opposition. The new Bill had passed its second reading in the House of Commons. Some further work had been done on the company's power scheme at Orsières in Switzerland, but operations had since been suspended pending more favourable times. The profits for the year were £348,108 (issued capital, with debentures, £2,637,189), and the directors recommended a dividend on the ordinary shares bringing the total dividend upon them for the year up to 10 per cent. The liquid resources of the company were satisfactory.

**NEW ISSUES.**—Lever Bros., Ltd. has made an issue of £4,000,000 seven per cent. first mortgage debenture stock at 92½ per cent., redeemable at par in 1941, or in 1931 at the option of the company. The authorised share capital of this company is £130,000,000, of which £46,996,499 has been issued; assets at December 31 last were valued at £50,734,770. The Mond Nickel Co., Ltd. has offered £1,300,000 of 8 per cent. mortgage debenture stock at 98, redeemable in 1941 at 104 per cent. The issue was quickly subscribed. An issue by the British Oxygen Co., Ltd. of £250,000 of 8 per cent. first mortgage debenture stock at 96 per cent. was also rapidly taken up.

## OFFICIAL TRADE INTELLIGENCE.

(From the Board of Trade Journal for March 24, 31, April 7.)

### OPENINGS FOR BRITISH TRADE.

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W. 1, from firms, agents or individuals who desire to represent U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number.

Locality of Firm or Agent.	Materials.	Reference Number.
Australia .. ..	Crockery, imitation leather ..	362
British West Indies ..	Glassware .. ..	426
Canada .. ..	Artificial silk yarns .. ..	393
" .. ..	Crockery, glass, scientific instruments, chemical plant, sugar machinery (catalogues of) ..	"
" .. ..	Drugs .. ..	4
" .. ..	Creosote .. ..	—
Cyprus .. ..	Fertilisers, soda crystals ..	427
New Zealand .. ..	China, glassware .. ..	398
Belgium .. ..	Pitch .. ..	372
Bulgaria .. ..	Creosote, zinc chloride ..	—
France .. ..	Asbestos materials, vulcanised fibre, emery cloth ..	402
Italy .. ..	Leather .. ..	403
" .. ..	Soap and paper-making materials, oils and starches ..	433
" .. ..	Oils and fats .. ..	434
Norway .. ..	Spring and tyre steel ..	378
" .. ..	Steel rails .. ..	379
" .. ..	Iron and steel sheets and pipes, steel wire ..	405
" .. ..	Pig iron, steel plates ..	436
Serbia .. ..	Creosote .. ..	—
Spain .. ..	Chemicals, drugs, paints ..	380
" .. ..	Iron and steel hoops ..	408
Sweden .. ..	Cocoa, sugar, syrup ..	382
" .. ..	Chemicals for textiles ..	383
Switzerland .. ..	Salt .. ..	438
" .. ..	Industrial porcelain, china clay, non-ferrous metals ..	439
Algeria .. ..	Chemical products, leather ..	441
China and Japan .. ..	Iron, steel .. ..	385
Eastern Siberia, Manchuria .. ..	Pottery, glassware, belting ..	386
United States .. ..	Rubber goods .. ..	442
Mexico .. ..	Chemicals .. ..	452
Mexico and Cuba .. ..	Pottery, soap .. ..	451
Panama .. ..	Drugs .. ..	453
Argentina .. ..	Paint, varnish .. ..	446
" .. ..	Chemicals .. ..	447
" .. ..	Rubber goods, metal sheets, tubes, etc. .. ..	449
Uruguay .. ..	Wire, ropes .. ..	391

\* H.M. Trade Commissioner, 257/260, Confederation Life Buildings, East Queen Street, Toronto, Canada.  
 † High Commissioner for Canada, 19, Victoria Street, London S.W. 1.

### TARIFF. CUSTOMS. EXCISE.

**Australia.**—As from April 1, licences for the import of foreign dyes, whether from stocks in the U.K. or not, are subject to permit issued by the High Commissioner's Office.

**Austria.**—Import licences are now required for certain leather and leather wares, and export licences for tanning materials, leather and leather wares (with some exceptions).

**Belgium.**—It is proposed to increase the customs duties on, *inter alia*, photographic apparatus, perfumes, pottery, glassware (except bottles), and mineral waters.

Licences are no longer required for the export of bottles, demijohns, cylinders for compressed and liquefied gases, carbonate of soda, natural cement, undressed hides and skins, superphosphates, linseed, rape-seed, scrap metals, coal-distillation products, and pyrites residues.

The export of bricks, artificial cement, raw bones, and fertilisers (except superphosphates) is subject to licence, but fertilisers, except potash salts, may be exported without licence as from June 1.

*Bulgaria.*—Export duties have been reduced on certain skins and edible fats.

*Federated Malay States.*—The export of china clay, felspar, and china stone is prohibited as from January 7.

*Fiji.*—The complete text of the revised customs tariff schedules may be seen at the Department, 18, Queen Anne's Gate, London, S.W. 1.

Exportation of gold and silver coin and bullion, rice, and raw sugar is prohibited as from December 15, 1920.

*France and Algeria.*—The export duty on oilcake has been modified.

The restrictions on the import of cellulose pulp and of paper in rolls have been withdrawn.

*Greece.*—The restrictions on the import of calcium carbide have been removed.

*Grenada.*—Additional export taxes have been levied on cocoa, cottonseed, nutmegs, and mace.

*Italy.*—Export licences are no longer required for manganese ore, magnesite bricks, newsprint paper, quinino salts, copper sulphate, and zinc waste.

Lead and zinc ores are again subject to export licence.

*Malta.*—The metric system of weights and measures will be used in levying customs duty as from July 1. Articles affected include alcohol, alcoholic beverages, malt, edible oils, petroleum, saccharin, sugar, spirit varnish, and vinegar.

*Martinique.*—The export duty on sugar has been increased to 21 fr. per 100 kg.

*Netherlands.*—The prohibition on the export of phosphatic fertilisers, glass powder, wood, zinc ore, and zinc oxide has been withdrawn.

The export of 240,000 metric tons of sugar-beet will be permitted, although the export prohibition remains in force.

Import duties have been increased on chloral hydrate, sulphuric and acetic ether, collodion, chloroform, spirit of nitrous ether, and similar substances prepared from or with alcohol.

*New Zealand.*—The regulations relating to the standards and labelling of vinegar are set out in the issue for April 7.

*Nigeria.*—Non-proprietary medicinal preparations containing alcohol imported by registered medical practitioners, dentists, and licensed druggists may be exempted from duty by the Governor in Council.

*Portugal.*—Paper fabrics and earthenware insulators now pay an increased customs duty.

*Rumania.*—The export duty has been modified on certain petroleum products and on oilcake.

*Serb-Croat-Slovene State.*—The export of all goods is allowed under conditions laid down in the Decree of April 16, 1920, with some exceptions, among which are sugar, gold, silver, and scrap iron.

*Spain.*—The export duty on oilcake has been abolished, but the export thereof is still subject to licence.

*Switzerland.*—Importation of industrial alcohol by private persons is prohibited.

As from March 18, import licences are required for paper, cardboard, and wares thereof, and for glass bottles.

Special export licences are required for, *inter alia*, cocoa powder, condensed milk, waste paper, scrap iron, copper turnings, precious metals, saccharin, potassium permanganate, indigo, and certain coal-tar colours.

*Tunis.*—Sugar may now be imported freely under certain specified conditions.

## GOVERNMENT ORDERS AND NOTICES.

### EMERGENCY REGULATIONS, 1921.

An Order in Council, issued on April 4, under the Royal Proclamation of Emergency, makes Regulations which empower the Government to take possession of land, works, plant, food, material, stores, etc., to regulate transport by road, light railway or canal, and requisition vehicles, to close ports and harbours, prohibit exports, control the supply of gas, water, electricity, and motor spirit, and to take possession of all or any coal mines, together with any plant, vehicles, railway wagons, etc., necessary for maintaining the supply and distribution of coal.

**COAL EMERGENCY DIRECTIONS.**—The Coal (Emergency) Directions, 1921, issued by the Secretary of Mines provide for a reduction of 50 per cent. in the industrial consumption of coal and fuel derived therefrom, prohibit the shipment of coal either as cargo or bunkers save with the permission of the Secretary for Mines or a person authorised by him, and regulate the supply and distribution of coal. Lighting used for advertisement and display is prohibited, gas and electricity companies are empowered to reduce the supply in order to economise coal, and the use of gas or electricity for power production is restricted by 50 per cent. The Directions came into force on April 2.

**PROHIBITED EXPORTS.**—Licences are no longer required for the export to Russia of any goods other than those the export of which is prohibited, except under licence, to all foreign countries.

## REVIEWS.

**LES ÉTHERS CELLULOSIQUES.** Pt. I.: *Les Éthers Minéraux de la Cellulose.* Vol. I.: *La Nitrocellulose et le Celluloid.* By ANDRÉ DUSOSC. Pp. 334. (Paris: A. D. Cillard, 1920.) Price 45 francs.

A book bearing this title naturally attracts the attention of celluloid chemists, explosives chemists, and that increasing number of scientists who are investigating the colloidal behaviour of nitrocellulose. It is to be feared that each class will feel some disappointment after reading the book. Celluloid chemists and explosives chemists will find, it is true, a number of full descriptions of plant and processes, but not much that is new, and they will occasionally find that the data relating to the two industries are difficult to disentangle. Colloid chemists, on the other hand, will find little that is of interest, except some generalities on the colloidal constitution of celluloid. Possibly later volumes will be more stimulating in this respect. Some most important researches on nitrocellulose which have a direct bearing on the constitution of celluloid have recently been published, and it is to be hoped that the author will discuss these in due course.

The present volume deals with the manufacture of celluloid as far as the preparation of bleached and washed nitrocellulose pulp. It opens with a historical introduction, in which the author accepts the account of the discovery of celluloid given by Chandler (*cf. J.*, 1914, 225) on the occasion of the presentation of the Perkin medal to J. W. Hyatt in 1914. This account led at the time to a somewhat acute discussion (*cf. J.*, 1914, 678) on the relative claims of Hyatt and Daniel Spill, in which neither side perhaps did justice to the versatile genius of Alexander Parkes; however, the claims of

Parkes to fame rest also on his researches in electro-deposition and metallurgy, and there is no need to reopen the discussion. The chapter on the physical and chemical properties of celluloid will prove the most interesting to those outside the industry, and here one is impressed with the absence of direct experimental evidence for the theories advanced. The truth is that the experimental difficulties are so great and the complications so many in the case of the concentrated plastic masses obtained in celluloid manufacture that the results obtained from experiments can only be empirical. It is usually impossible to control experiments so that only one factor varies at a time, which is the ideal condition sought for in research. How many chemists would be prepared to assert that any two samples of nitrocellulose were identical? This difficulty arises at the outset, and since the nitrocellulose must be bleached, pulped, washed, dried, blended with solvents, kneaded, rolled, pressed, sliced, and stove, and since, moreover, a few minutes more or less spent in some of these processes may profoundly modify the properties of the product, it is frequently impossible to ascribe the difference between any two samples of celluloid to a single cause. We are forced to conclude that the systematic study of solid celluloid must start with dilute solutions of nitrocellulose, and, although the author's views on the colloidal phenomena accompanying celluloid manufacture are interesting, we think that, for the present at any rate, they will prove sterile. A more promising system of attack would be to examine Bingham's division of colloid solutions into viscous liquids and highly mobile plastic solids, and to investigate nitrocellulose solutions in varying concentrations from this point of view. Until we know more about the distribution of solvent between the phases in dilute nitrocellulose solutions, we cannot expect to understand much about the constitution of solid celluloid.

The raw materials of celluloid manufacture are treated fully, and the author has made good use of the information contained in papers read before this Society by de Moseenthal (*cf. J.*, 1904, 292; 1911, 732). The chapter on nitration is preceded by a theoretical discussion on etherification, which, although not directly applicable to the nitration of cellulose at present, is perhaps a useful caution against too much rule-of-thumb in the control of the factory operation. The somewhat obvious mathematical treatments of nitration and doping due to Clément, which have already been published more than once, are less useful than Fowler's graphical method (*cf. J.*, 1919, 347), and might have been omitted, as well as some of the ancient formulae dating back to the infancy of the industry. There is an interesting section on the Thomson displacement process in which the author sums up strongly against its applicability to the manufacture of nitrocellulose intended for celluloid manufacture. The last two chapters deal with stabilisation, washing, pulping, and bleaching, and due emphasis is placed on the necessity for using pure water and for avoiding any process which will injuriously affect the colour of the product. This important distinction between explosives practice and celluloid practice is not always appreciated.

There are no illustrations of plant in the book, which detracts from its usefulness. The type is good and only a few misprints have been encountered, among which "Van T. Hoff" has a quaintly American flavour. It is to be hoped that in the succeeding volumes M. Dubosc will break away still further from the tradition that books on nitrocellulose should be merely abstracts of patents of varying vitality, and will discuss the problems of celluloid manufacture in the light of his own unquestioned experience.

F. SPROXTON.

**INDIAN TANSTUFFS.** By J. A. PILGRIM, *Government of India, Board of Industries and Munitions.* (Calcutta: Superintendent Government Printing, India, 1920.) Price 6 annas.

The Government of India is to be congratulated on the production of a short, concise report of valuable chemical research in which the practical and commercial application of results is not overlooked. Mr. Pilgrim's handbook contains the results up to April, 1920, of his and Mr. W. Fraymouth's examination of the barks, leaves, fruit, etc. of Indian forest species, mainly from the point of view of manufacture of tannin extracts for export. The requirements of the Indian tanner, *i.e.*, cheap tanstuffs to be used in direct contact with the hide, are also not lost sight of. With the exception of one or two species, which are duly noted, many samples of each tanstuff have been examined, and it is evident that the sampling of each bark or leaf has been carried out with great care, thus greatly increasing the value of the conclusions arrived at. Hide-powder estimation of tannin is not an exact analysis, but if used as Mr. Pilgrim evidently uses it, it is of great value in determining the tannin value of barks, etc. We consider the publication of negative results, *e.g.*, those on pages 24 and 25, as of great value. It is not always appreciated that negative results are as valuable as positive results in research work.

Analyses and practical leather-making have established the value of Thawai (*Woodfordia floribunda*) bark, and if this could be produced at a low price in quantities, it is clear that it would threaten the consumption of South African wattle. It is to be regretted that the present bulletin does not contain figures of cost of the tanstuffs as the earlier bulletin did. It would be best to express these in shillings per unit of tannin per ton, free on rail in India. This applies also to a recent report in a trade paper showing extraordinarily favourable figures of tannin and non-tans in certain oak and chestnut barks which have been studied in Northern Burma (presumably by Mr. Pilgrim). Nothing whatever is said of the distance from the rail at which the forests of these trees are found, nor whether labour is available at reasonable prices for collecting the barks. Mr. Pilgrim continues the practice of examining samples of bark from each part of the tree, and we would urge that a full range, from bole, low branches, high branches, twigs, etc., should be studied in every case where the first examination demonstrates the possibility of a valuable source of tannin.

The principle adopted in the earlier researches at Maihar, *viz.*, to collect the tanstuff without destruction of the tree, appears to have been less rigorously followed in the later work recorded in this report. The history of the tanner's bark has always been the same. A cheap and good bark becomes expensive as the demand grows and as the most accessible forests are depleted. It was so with English oak, with hemlock, and now with South African wattle, although in this case the bark exploiters have replanted vigorously. It appears probable that, other conditions being equal, the bark which can be got without destroying the trees will finally hold the market.

In a thoughtful preface Mr. Pilgrim explains his principles in studying Indian tannins from the point of view of extract-making. Unfortunately he adopts the standard of "crystals." It is a fact that all the biggest tanners, in Britain at any rate, if they use solid extract at all, demand a clean-fracture solid which generally contains more moisture than the 5 per cent. carried in "crystals."

The extent and resources of the forests of the Indian Empire are as yet little known in Britain, but now that the Forest Research Institute at Dehra Dun has received bigger grants and has engaged several new scientific officers, it is to be

expected that regular reports on the progress of research will appear. It is to be hoped that after the present trade depression is over, British financiers will study the work done and take the earliest opportunity of exploiting new discoveries. During the years before the war a large number of German firms in India had commenced such exploitation. The opportunity presented now by the absence from India of all ex-enemies will not last for ever, and the presence in India of other than British firms should be closely watched, if the spirit of the new reforms, i.e., close co-operation between the Britisher and the Indian, is to be followed.

TEXT BOOK OF CHEMICAL ENGINEERING. By EDWARD HART. Pp. 211, with 200 figures. (Easton, Pa.: The Chemical Publishing Co.; London: Williams and Norgate. 1920.) Price 4 dollars.

There are not many text-books of chemical engineering, and therefore many chemists will welcome the opportunity of reading this small but attractive book, which is printed in good style, with profuse illustrations. The reading matter is, on the whole, very good, but all too brief to be of great service to the chemical engineer. As stated by the author, the book is primarily intended for the use of the college student and purposes to give him a general idea of the type of plant employed for standard operations in the chemical industry. Comparisons of one system with another are seldom attempted; these, though apt to be invidious, are very helpful. Exposition of the theory underlying the design or operation of the plant is also lacking. Historical matter is curtailed to a minimum and old-fashioned, inefficient appliances are omitted. For this reason the book gains in conciseness and can be said to be quite up-to-date as regards the subjects treated.

The chapters on crushing, filtration and drying are specially good, except for the lack of critical comparisons. Probably most industrial chemists could regard the chapters on boilers, prime movers and plumbing as superfluous, though the college student will, no doubt, find them useful.

There appear to be few errors of commission, but, naturally, a good many of omission. We do not find, for example, any information with regard to water, gas and electricity supply, disposal of waste, centrifugal pumps, electrostatic precipitation, furnaces, gas washing, recovery of light spirit, fractional condensation, refrigeration, liquefaction, heat exchange, etc.

Whilst the general quality of the illustrations, which fill more than half the pages, is excellent, a few are distinctly open to criticism. For example, the circular stoneware tower appearing as a full-page illustration on page 63 (and again on page 201) shows acute and right-angled pipe-bends, which the engineer tries to avoid. Some of the illustrations, for example, figs. 188 and 189, are not accompanied by the necessary descriptive and explanatory text. Fig. 154 is badly drawn.

As a short and handy book for students this work has much to commend it.

W. B. DAVIDSON.

CORRIGENDA.—In the issue for March 31, p. 113 R, under Legal Intelligence, Rail Conveyance Classification of a Washing Powder, substitute "Class C" for "Class 2" in lines 8, 15, and 26; and in lieu of "soap powder" read "washing powder" in lines 11 and 24.

## OBITUARY.

### LORD MOULTON OF BANK.

By the death on the 9th ultimo of Lord Moulton the Society has lost one of its most brilliant members. From New Kingswood School, Bath, John Fletcher Moulton went to Cambridge (St. John's College), where he became Senior Wrangler and First Smith's Prizeman. He took his M.A. degree of London University, and later that of Cambridge, becoming soon after a Fellow of Christ's College. Resigning his Fellowship in 1873, he was called to the Bar in the following year, taking silk in 1885. As the most successful patent lawyer of his day, he acquired knowledge of the arts of the chemist, the physicist, and the technologist, so that he had understanding of their language and their writings, a possession of special importance in handling the great number of legal cases pertaining to industry, and especially chemical industry, in which he was engaged. He was a member of the House of Commons, a Lord Justice of Appeal, a Lord of Appeal, and a Privy Councillor.

Here, in all probability, this notice would have concluded had it been written before August, 1914.

But war came, and Lord Moulton, then 70 years of age, placed his talents and his energies at the disposal of his country in her hour of trial. He first took up the matter of dye production—German supplies having suddenly ceased, leaving the textile industries faced with the impossibility of finishing their productions—the politicians' cry of "Business as usual" ringing more clearly in the ears of the nation than the danger alarm raised by the few who knew Germany and German aims. But it was quickly realised that the country was confronted with more serious tasks than the dyeing of cottons and silks, and, putting dyes for the time being on one side, Lord Moulton was entrusted with the chairmanship of a small advisory committee on high explosives. At that time the British Government made propellants but no high explosives, except a little tetryl (trinitrophenylmethyl nitroamine) at Waltham Abbey. Picric acid, the standard one, was purchased from a limited number of contractors in relatively small quantities. TNT had hardly come upon the scene; its manufacture from toluol had been proposed to the writer some time before the outbreak of war, but in response to his inquiry as to the attitude of the War Office, he was told it was not favoured, although it had been brought into use in Germany. In England it was used only for minor purposes and in very small quantities—practically as a by-product of the dye industry.

The nation's pitiable plight of unpreparedness both in raw material supplies and in large scale processes soon became increasingly apparent. Germany had monopolised the organic chemical industry so completely that the greatest difficulties were experienced in this country when she was suddenly called upon to produce nitro-compounds in large quantities. We were short of plant, of trained chemists, and of manufacturing knowledge. Lord Moulton realised, more than any man, the inadequacy of our resources and the magnitude of our requirements. As chairman of the High Explosives Committee (A.6) under the Director of Artillery, his powers were gradually extended until in February, 1915, he had liberty to utilise to the utmost the capacity of the country. Four months later he became Director-General of the Department of Explosives Supply in the newly formed Ministry of Munitions of War. In order to work out the chemical and technical problems involved in manufacture on the necessary scale, the whole chemical industry throughout the length and breadth of the land was organised and co-ordinated



under his direction, no undertaking being too small to be entrusted with a share of the task. The production of the necessary raw materials was similarly dealt with, and before many months passed both crude- and final-product manufacturers were working in friendly consultation and competition, inspired by a most efficient system of costing and yield-checking worked out and put into operation at the headquarters of the department.

The preparation of high explosives was beset with greater difficulties than that of propellants, for besides the Government factory several private firms were well versed in the manufacture of cordite. On the other hand, the resources for picric acid (the standard high explosive) were inadequate not only as regards manufacturing capacity but also as regards raw material. This led to the manufacture of synthetic phenol and the inauguration of the production of TNT. As neither of these substances could be turned out in quantities sufficient to satisfy shell-filling requirements, Lord Moulton urged the addition of ammonium nitrate explosives, such as ammonal, schneiderite, and, later, amatol. Numerous problems of filling, detonation, and exploding were thereby introduced which required close investigation. Ultimately all troubles were overcome and the mixed explosives, in the preparation of which enormous quantities of ammonium nitrate were required, were entirely successful. The difficulties besetting adequate production were increased by the high standard of purity necessary for Service purposes, and the specifications were being constantly revised in order to reconcile quality and quantity. It has already been stated that the manufacture of TNT was practically new in this country, and Lord Moulton played a large part in raising it from small beginnings to a very large scale. This needed the organisation of materials, methods, and plant, with which he kept in close touch and in the development of which he rendered great assistance. Originally slow and wasteful, the processes were ultimately improved until they showed an efficiency upon which it would be difficult to improve. Purification methods were of great importance. These were originally attached to each plant, but one or two accidents led to their removal to separate works. The original process was crystallisation from alcohol, afterwards abandoned for washing in the cold first by alcohol, and later with sulphite. The latter method was introduced by Lord Moulton from France, where he found it in use. Safety precautions was another matter requiring careful watching, and Lord Moulton organised a special "Safety of Factories Branch" for the independent inspection of the many chemical factories. The fixation of nitrogen did not mature during the war. It formed the subject of investigation by a separate body, the Nitrogen Products Committee, of which Lord Moulton was a member. He several times expressed the opinion that one of the chief determining factors which induced Germany to choose August, 1914, as the date for launching the war was the perfecting and extension of the Haber process—without synthetic nitrates she would have lost the war within a year or less. Our own dependence upon imported nitrates was one of the weakest links of our defence, and the destruction of ships carrying them caused great anxiety and at times acute shortage.

In the spring of 1918 Lord Moulton took up the supply of poison gases, and this necessitated the establishment of factories for their production. The work was very distasteful to him, but he prosecuted it with vigour and entered closely into all the chemical and technical problems involved, which were naturally very numerous. The chief products were dichloro-diethylsulphide, phosgene, chloropicrin, and some arsenic compounds.

Lord Moulton was an energetic advocate of the establishment of a British dye industry on a scale large enough and strong enough to stand up against German competition, and he became Government representative and chairman of British Dyestuffs Corporation, Ltd., in 1919, holding the position until the following year, when he returned to his judicial labours.

It is not too much to say that under him the whole chemical industry of the country was not only reorganised on a basis enabling it to fulfil its war-time tasks, but also, and only second in importance, to meet adequately the post-war requirements of the nation. No man was better able to undertake two tasks of such magnitude. He was not merely an able lawyer, but a far-seeing and cultured man of the world and its affairs; and among other qualifications the possession of a large fortune acquired at the Bar must not be overlooked, since it placed him in a position of unassailable security in dealing with Governmental methods and red tape which might have weighed against the success of one less prominently and assuredly circumstanced. His name will be indissolubly connected with the future of chemical industry in Great Britain.

## PUBLICATIONS RECEIVED.

CHEMICAL TECHNOLOGY AND ANALYSIS OF OILS, FATS AND WAXES. VOL. I. By J. LEWKOWITSCH. Edited by G. H. WARBURTON. Sixth edition, entirely rewritten and enlarged. Pp. 682. (London: Macmillan and Co., Ltd. 1921.) Price 36s.

THE ELECTRONIC CONCEPTION OF VALENCE AND THE CONSTITUTION OF BENZENE. By H. S. FRY. Monographs on Inorganic and Physical Chemistry, edited by A. FENLAY. Pp. 300. (London: Longmans, Green and Co. 1921.) Price 16s.

MODERN CHEMISTRY, PURE AND APPLIED. By A. J. HALE. Six Volumes. Vol. I., pp. 272; Vol. II., pp. 276. (London: Virtue and Co., Ltd. 1921.) Price 16s. per volume.

TRAITÉ DE LA CONSERVATION ET DE L'AMÉLIORATION DES BOIS. By M. DE KEGHEL. *Encyclopédie Industrielle*. Pp. 360. (Paris: J.-B. Baillière et Fils. 1921.) Price 10 fr., bound 15 fr.

DIE WELT DER VERNACHLÄSSIGTEN DIMENSIONEN. By W. OSTWALD. Fifth and sixth editions, revised. Pp. 253. (Dresden and Leipzig: Theodor Steinkopff. 1921.) Price 7s.

GRUNDRISS DER KOLLOIDCHEMIE. PART I. By W. OSTWALD. Sixth edition, forming a reprint of the fifth edition. Pp. 253. (Dresden and Leipzig: Theodor Steinkopff. 1921.) Price 10s.

KLEINES PRAKTIKUM DER KOLLOIDCHEMIE. By W. OSTWALD, assisted by P. WOLSKI. Second edition. Pp. 159. (Dresden and Leipzig: Theodor Steinkopff. 1921.) Price 4s.

IMPERIAL INSTITUTE MONOGRAPHS WITH SPECIAL REFERENCE TO THE BRITISH EMPIRE. (London: John Murray. 1920 and 1921):—

COAL. By J. H. RONALDSON. Pp. 166. Price 6s.

CHROMIUM ORE. By W. G. RUMBOLD. Pp. 38. Price 3s. 6d.

PUBLICATIONS OF THE UNITED STATES GEOLOGICAL SURVEY. Department of the Interior. (Washington: Government Printing Office):—

MAGNESITE IN 1919. By C. G. YALE and R. W. STONE.

PHOSPHATE ROCK IN 1919. By R. W. STONE.

SAND-LIME BRICK IN 1919. By J. MIDDLETON.



## TOWARDS UNITY.

It is encouraging to note that the various proposals relating to the organisation of chemistry in this country which have been put forward from time to time by the Federal Council for Pure and Applied Chemistry have met with a uniformly favourable reception, and that the only criticism heard is that action has been too long delayed. In other words, to use Matthew Arnold's phrase, appreciation has been the soul of criticism, and this circumstance affords a very happy augury of benefits to come.

An important move in the direction of unity was made by the Council of the Society of Chemical Industry at its last meeting, when, in response to a suggestion received from the Council of the Chemical Society, it was resolved unanimously that the existing machinery of our Local Sections should be made available to a certain extent to members of the Chemical Society, the Society of Public Analysts, and, by implication, to members of other chemical organisations. The specific suggestion referred to was that papers on pure chemistry, whether contributed by members of the Society of Chemical Industry or others, should be accepted for reading at the meetings of our Local Sections, and that the Chemical Society should consider for publication any such papers sent on to it by us. The Council of this Society, in approving the suggestion as a council, decided to recommend its adoption to the Local Sections, and to leave details to be settled later.

The advantages to be derived from such a scheme are many. For some time past there has been a congestion of papers presented for reading at the monthly meetings of the Chemical Society, with the result that some of them are not read at all, and others are inadequately discussed. Furthermore, many Fellows are put to considerable inconvenience and expense in travelling to London to read their papers, and by increasing the number of localities where and occasions when papers can be read, delay in presentation and publication will be obviated, and a greater proportion of the contributions will be discussed and criticised. A paper, when read, may give rise to instructive and constructive criticism, valuable alike to author and audience, even if it be not sufficiently important to warrant subsequent publication; and the mere fact of public presentation would ensure, at any rate, a notice of the paper in the chemical scientific press, and perhaps also in the local newspapers.

From the point of view of our Local Sections it would appear that the reading of papers on pure chemistry would add to the attractiveness of meetings by providing a greater variety of subject matter. Those Sections which sometimes experience difficulty in filling their programmes would find relief in the greater quantity of material available, a consideration which applies more particularly to University towns; and other Sections which, owing to favourable environmental or other circumstances, have no such difficulty would reap the advantages of better attendances and wider publicity. In such cases it may be that the time devoted to the reading of each paper on the agenda would have to be curtailed, but many would regard this as an advantage; long papers are apt to become wearisome, and a free, fluent delivery of "things that matter" is preferable to reading verbatim, especially if an abstract has been circulated beforehand. Further, such composite gatherings would afford a common meeting-place for chemists of all classes and descriptions; readers of papers on technical chemistry would have the advantage of criticism by those who cultivate the "pure" science, and

*vice versa*; and the opportunities afforded for social intercourse would be generally fruitful of much good, alike from the scientific, technical, and professional points of view.

But above and beyond all is the consideration that the whole-hearted adoption of the proposal would give impetus to the movement for securing that unity in the profession and practice of chemistry which many of us have so much at heart. The advantages accruing from co-operation and co-ordination have been dinned into our ears in recent years almost *ad nauseam*, and the time is surely ripe for action. Although the realisation of certain other proposals, such as that for a central Chemical House, may have to be postponed owing to the exceptionally adverse industrial conditions, there is much that can be done without delay. Bricks and mortar alone cannot make a united profession; it is the spirit that counts; and in the decision to extend to other chemists certain facilities which have hitherto been restricted to our own members, we see a manifestation of that desire for unity which alone can lead to the end we have in view.

## DANGEROUS DRUGS.

Probably very few chemists see the pharmaceutical and medical press, and those who do are not likely to have read with care the articles which have appeared there recently on the draft regulations made by the Home Office under the Dangerous Drugs Act. These regulations are, however, likely to prove very detrimental to the interests of professional and research chemists. Thus Article 2 prohibits the manufacture of morphine, cocaine, ecgonine, or diamorphine, or their respective salts, or medicinal opium, except by persons properly licensed or authorised to do so, working on premises licensed for the purpose and in accordance with the conditions of such licence or authority. "Manufacture" is nowhere defined in the regulations, and it is a moot point whether a research chemist effecting a new synthesis of one of these alkaloids, or an analytical chemist determining morphine by extracting it from a plant or from the stomach contents of a poisoned animal, would not be "manufacturing" the alkaloid and so infringing the regulations, unless he takes the precaution to acquire a licence for himself and his premises.

Similarly in regard to Article 3, a chemist who effects a synthesis of one of these alkaloids or prepares one of them from a new source will be infringing the regulations if he sends some of his material to a colleague for confirmatory or supplementary tests, or even for inspection, unless he duly acquires a licence to supply the drug and acts in accordance with the terms of such licence or authority. But in this case not only is he himself likely to get into trouble, but the recipient may, unless duly licensed, be in the unfortunate position of having wrongfully secured possession of the alkaloid in question unless he, too, has taken the precaution to secure beforehand the necessary licence or authority. Experience gained hitherto in the working of the Cocaine Regulations shows that a long and tedious delay precedes the granting of a licence, so that a chemist requiring one of the so-called dangerous drugs may in future find that it takes many weeks to obtain even the smallest quantity of them. It also appears that a chemist who has acquired a supply of any of these drugs even for research or other professional purposes and has unwittingly left a grain or two in a bottle in some dusty corner of a stock-cupboard, or has

knowingly kept some for purposes of reference or comparison, will either have to destroy it or acquire a licence or other authority to retain it, and if he retains it he may apparently have to produce it when required for inspection by a constable or other duly accredited representative of the Home Office. As for purchasing, keeping and using these drugs for professional purposes, most chemists will think twice before undertaking to keep in order the licences, forms, books, etc., necessary to secure him immunity from the pains and penalties likely to overtake anyone undertaking such an operation.

These are a few of the possible difficulties which will occur to a chemist who casually peruses the draft regulations. The catalogue would no doubt be of considerable length if they were considered with a solicitor, versed in the possible legal twists and turns of an apparently innocent sentence in any of the 15 articles. Matters are apparently somewhat simpler if the chemist happens to be in addition a ship's captain with a ship at his disposal, a registered pharmacist, medical practitioner, dentist or veterinary surgeon; and the professional chemist who has much to do with these drugs, dangerous in more senses than one, will probably find his best course to be to take a medical degree, although even then he will probably need the help of a solicitor—unless he becomes that also—to steer clear of the difficulties which his professional duties may thrust upon him.

Fortunately the regulations are still only in draft, and it is earnestly to be hoped that the Institute of Chemistry and the Society of Public Analysts, which are the bodies best qualified to speak for chemists on such a matter, will promptly call the attention of the Home Office to a fact which it appears to have forgotten, namely, that professional, as well as legal, chemists, occasionally have to deal with these dangerous drugs in the course of their work. A short addition to the regulations indicating that chemists engaged in research, teaching or professional practice are exempt from the general restrictions would apparently meet the necessities of the case.

## DISEASES OF OCCUPATION.\*

STEPHEN MIALI.

The human body was designed for use in circumstances widely different from those which exist in modern industrial areas, and though evolution can accomplish much it is slow in its operation, and many thousands of years are required for any appreciable change in the qualities of the human organism. As chemical industry becomes more and more complicated it becomes more and more specialised, and we find now men and women who for the whole of their working life are engaged in the same processes, involving exposure to the same chemical compound hour after hour, day after day, and year after year. Whether this is entirely necessary I am not sure, but I wish to indicate very briefly some of the dangers of this sort of life and how they may be prevented or reduced to a minimum.

Broadly speaking, one may say that constant exposure to any chemical compound is likely to be injurious to health, particularly if the compound is likely to give rise to dust, fumes, or gas, and that an abundant supply of fresh air is, as one might expect, the best method of protecting the health of the workers. No doubt some compounds are worse than others, but one may say that all

forms of dust and fumes are bad. Constant exposure to coal dust and tar causes what is known as pitch-makers' cancer, the tar being in this case the main irritant; the manufacture of marmalade causes injury to the skin by reason of the peeling of oranges and lemons; miners exposed to coal dust or to a fine silica dust have their lungs affected in a particular way; and workers exposed to dust of metallic compounds, phosphorus fumes, etc., contract specific kinds of poisoning.

Carbonic-oxide poisoning, carbon-dioxide poisoning, poisoning by chlorine, nitrous fumes, ammonia, aniline, tetrachlorethane, turpentine, ether, and acetone are not uncommon in industrial conditions.

The diagnosis of these diseases is often difficult, the cure is often difficult, the conditions under which they are contracted differ enormously, but they are usually all preventible by good ventilation.

The incidence of phosphorus poisoning is well known. Five-and-twenty years ago there were five or six cases a year in this country of phosphorus poisoning out of about 1700 persons employed in phosphorus processes. This has now been reduced to about three cases every two years, and the cause of the reduction is well known: to a large extent red phosphorus is employed instead of yellow phosphorus. The Swiss Government in 1879 introduced a Bill for the prohibition of the use of yellow phosphorus, which in due course became law, but it had to be radically altered because the disease became more prevalent. It seems that the workers imagined that when yellow phosphorus was prohibited they could dispense with a number of precautions which had usually been adopted, and they engaged to a considerable extent in processes where red phosphorus was used under very unhygienic conditions. Finally, the Act was practically repealed, and regulations were adopted by the various cantons which ensured suitable hygienic conditions of work. In France sesqui-sulphide of phosphorus, which gives off no fumes, has for many years been used instead of yellow phosphorus, and phosphorus poisoning has been stamped out in that country. It is interesting to note that whereas good ventilation and the ordinary rules of hygiene have in the main been responsible for the diminution of phosphorus poisoning in this and other countries, the substitution of some other forms of poisoning has been repeatedly advocated; in America there was a practice in match factories of hanging round the necks of the workers small vessels containing turpentine. In Italy a non-poisonous match was made of which the principal ingredient was persulphocyanic acid. The true remedy is a simple and efficacious one. Sir Thomas Oliver, who was a member of the Phosphorus Commission which reported rather more than 20 years ago, and is a recognised authority on dangerous trades, states in relation to phosphorus poisoning: "Of all hygienic measures full and free ventilation is the best."<sup>1</sup>

Cases of poisoning by carbon monoxide, nitrous fumes, ammonia, and other gases are on the increase in this country; there were 90 reported cases in 1913, and 132 cases in 1919; of these 59 in 1913 and 85 in 1919 were cases of carbonic oxide poisoning. Almost all these cases were due to ignorance on the part of the workers of the dangerous character of the fumes, or carelessness. There is no difficult chemical problem involved in the prevention of this form of poisoning; common sense in preventing and detecting leaks in flues will reduce such cases speedily, and workmen should be warned that products of combustion of all kinds are liable to be dangerous to health. A fair number of the cases of poisoning by such gases occur not in a closed room, but out of doors, in a gas works or lime kiln, or plant of that type. All these are preventible. In mines compressed air is largely used for drilling and for ventilation, and a number of deaths have

\* From a paper read before the Liverpool Section on January 24, 1921.

resulted from the overheating of the air compressor and the firing of the oil used for lubricating the cylinders.<sup>2</sup> During the war there were many cases of carbon-monoxide poisoning from the exhaust of small power petrol-engines installed in dug-outs or similar places, and there have been cases of poisoning from the exhaust attached to closed motor cars, motor lorries, and ambulances.

During the last two or three years cases of chronic benzene poisoning have been notified in this country which have resulted from the employment of benzene as a solvent for rubber, and one case is of exceptional interest. In May, 1918, structural alterations were made in a particular factory, wooden uprights being inserted in certain windows so that they could not be widely opened. In July, 1918, a workman employed in the room was reported as suffering from benzene poisoning, and the case proved fatal.<sup>4</sup> Analyses of the air in different parts of the room showed from two to ten parts of benzene in 10,000 parts of air. The ventilation of the room was improved and no further cases occurred.

Poisoning by turpentine vapour is a problem which has been recently investigated in connexion with the paint industry. It has been long known that many persons are particularly susceptible to the smell of a newly-painted room; the white lead, the turpentine, the linseed oil, and the dryers have all been suspected. I suppose no scientific man now believes in a volatile lead compound given off as a gas or emanation from lead paint, and experiments made during the last twenty years clearly point to turpentine as being the delinquent. Lehmann in 1899 described the symptoms produced in cats by turpentine vapour, and Sir Kenneth Goadby has more recently shown that the poisonous effects produced in animals by exposing them to the vapour given off by painted surfaces are caused by the turpentine content of the paint and not by the linseed oil, dryers, or metallic salt of the paint. Turpentine poisoning produces kidney disease, and seems to cause, or predispose to, gout. Sir Kenneth Goadby states in the joint book by Dr. T. M. Legge and himself<sup>5</sup> that, according to Garrod, gout is constantly occurring among painters.

Dr. G. Arbour Stephens, in a recent paper,<sup>6</sup> reports that during the last six years he has had eight cases where the analysis of the liver showed from about 0.1 to about 0.9 grains of cadmium per pound, with merely a trace of lead. It is presumed from the details he gives that these cases occurred in the zinc-smelting industry in South Wales, and that they were all fatal cases. The symptoms of cadmium poisoning include kidney disease, constipation, and loss of appetite. Zinc is known to be a general protoplasmic poison and, according to Harnack, in the higher animals it causes muscular paralysis and kidney disease. Copper, tin, nickel, and manganese have injurious properties, but the cases of poisoning by these are few and far between.

White lead has been manufactured here since at least the time of Queen Elizabeth, and a variety of lead compounds has been made for a century; lead or lead compounds are used in a remarkably large number of industries and processes, and it is not surprising that by the time the disease had been identified and the method of preventing lead poisoning had become known it had become very prevalent; indeed, as we look back, it seems appalling to think that thirty years ago or less there were in Europe hundreds, or even thousands, of cases of lead poisoning every year, and most of them easily preventable. In many towns lead poisoning was in those days contracted through drinking water kept in contact with lead; in the white lead trade there were in this country nearly 400 cases a year; in the china and earthenware trade there were upwards of 200 cases a year; in smelting, in brass works, in

printing, file cutting, glass cutting, shipbuilding and other trades there were over 400 cases, making a total of over 1000 cases per annum.

The attention of the public and of the Home Office was directed to the prevalence of lead poisoning at the end of the last century, when many inquiries were held and considerable information collected. The prohibition of the use of lead compounds in the pottery industry was threatened and various substitutes for lead were suggested. Finally regulations were put into force compelling the removal of dust, the wearing of overalls, regular medical examination, and so on. At the same time the manufacturers of china and earthenware were encouraged to use for glazing purposes an insoluble silicate of lead instead of the very soluble and very dusty white lead formerly used. On the whole the insoluble silicate has been a great boon to the manufacturers of pottery. It has been as satisfactory and cheap to use as the soluble white lead, more uniform in its composition and better to handle, and since its introduction the number of cases of lead poisoning in the china and earthenware trade has steadily declined. It came down from 200 a year in 1900 to 76 a year ten years ago, 15 cases in 1917, 11 cases in 1918 and 21 cases in 1919. The amount of lead in use in the industry has, so far as I am aware, not materially altered; the number of persons employed in the industry has slightly increased and the number of cases has declined to something less than one-tenth.

In the white-lead industry the diminution of cases has been somewhat similar, but even more marked: the number of cases was 399 in 1899, 189 in 1901, 40 in 1915, 18 in 1916, 17 in 1917 and none in 1918. The amount of white lead manufactured in England has declined during that period by about 10 per cent. and the number of persons employed is probably about the same as it was. These striking results have been attained by a careful study of lead poisoning during the last 20 or 25 years. Sir Edward Thorpe, Sir Thomas Oliver, and Dr. Legge, the Chief Medical Inspector of the Home Office, have been largely responsible for these results, and they were assisted in their efforts by a number of Home Secretaries, Inspectors of Factories and private individuals of both sexes. What is the vital factor necessary to the prevention of lead poisoning? Dr. Legge tells us in the Home Office Report for 1918,<sup>7</sup> and these are his words: "On the practical side little more is to be learnt as to how lead poisoning is caused, and it can be taken as axiomatic that all risk lies in inhalation of dust and fumes. These removed and prevented, there will be no lead poisoning." And again, in the Home Office Report for the year 1919,<sup>8</sup> Dr. Legge stated, "that locally applied exhaust ventilation is the sheet-anchor in the protection of the workers from lead dust and fumes and that these alone are the causative agents." With this knowledge it should be possible to reduce yet further the cases of lead poisoning in this country, and the next trade in which a serious effort is to be made is the painters' trade—I mean the painting of houses, ships, bridges and so forth, not the manufacture of paints and varnishes.

It is somewhat unexpected that lead poisoning should be caused not by the particles of lead swallowed by the worker, but by even more minute particles of lead compounds floating in the air and getting into his lungs and there acted on by the juices of the body and so getting into the system. But that this is the usual cause is now generally accepted.

The problem is beset with small difficulties which nevertheless are capable of being surmounted. To begin with, lead poisoning is in many cases by no means easy to identify. A doctor who regularly examines men who are employed in a lead factory

can, with a good deal of certainty, state whether a particular man is contracting lead poisoning and should be suspended from work or given employment which does not involve contact with lead compounds. But if a worker goes to the average careful general practitioner and complains of headache, colic, constipation, kidney trouble, and so on, it is pretty nearly impossible for the cause of these symptoms to be identified. There are so many things which will bring about these symptoms. Legge and Goadby—and I quote these as the greatest living authorities on lead poisoning—state in their book: "The headache complained of by painters is probably not due to lead poisoning but, as has been suggested, to turpentine." Turpentine, too, is probably the cause of the gout to which painters are subject. But of one thing we may be certain, if a man goes to a general practitioner and complains of such symptoms as I have mentioned, and states that he is a painter or employed in a lead works, his case will be duly reported as one of lead poisoning. This is not merely an opinion of my own. Legge and Goadby<sup>19</sup> say that there is "an increasing inclination to attribute chronic nephritis and even (without sufficient justification in our opinion) phthisis and pneumonia to lead poisoning on the death certificates of lead workers." I do not deny that lead poisoning exists among painters and others, but I am quite certain that a great many of the so-called cases of lead poisoning in the paint trade and in other trades have no connexion with lead at all. The cases of cadmium poisoning I have already mentioned were some of them certainly, and all of them probably, returned in the statistics as lead poisoning, and no one can say how many cases of turpentine poisoning, indigestion, Bright's disease, etc., among lead workers have been returned as lead poisoning, but the number must be very large.

From time to time proposals have been made to reduce the frequency of lead poisoning by prohibiting the use of white lead in paint, and this question will be considered at the next conference of the International Labour Office at Geneva. I want you as scientific men to consider somewhat carefully what such prohibition means; if the principle is once admitted, we may have to face the prohibition in their own industries of mercury, antimony, copper, arsenic, and a host of other chemicals which are not intended for human consumption. The prohibition of lead in paints only deals with a portion of the problem, and will give a false sense of security to the painters similar to that which operated so dangerously in the phosphorus workers in Switzerland. The proper solution of the problem is that which has been found so efficacious in many other diseases of occupation—fresh air and the removal of dust and fumes. It has been seriously advocated that to avoid lead poisoning houses should be painted with antimony compounds, titanium compounds, and other heavy metal derivatives; the fact is that the dust of nearly all compounds of heavy metals is injurious and that the fumes of nearly all volatile solvents are bad for the health.

It is known to all in the trade that the dust is produced in the dry rubbing-down of old paint by pumice-stone or sandpaper, and, in my opinion, this should be prohibited by law. Rubbing down can be done wet—perhaps not quite so easily, but sufficiently so—and the painters ought to be protected from exposure to dust of old paint.

It is possible to do away with the dust of the rubbing down by keeping the sandpaper moistened with a cheap mineral oil. The sandpaper lasts as well as without the oil and the result upon the paint is fully as good. This is a method with which many German painters are familiar, and if it could be generally introduced into this country a great

step forward would have been taken in improving the conditions in house painting and carriage and railway coach painting and ship painting.

Again, there is no reason why painters and those who prepare the paints for them should ever handle or be supplied with dry white lead or any other pigment; these should invariably be supplied ground in oil or some other medium. As to the vapours from the solvents in ready-mixed paints, I saw workmen in America a year or so ago painting the inside of a house by means of a spray, and attached to the painter was a tube communicating with the outside of the house enabling him to breathe fresh air instead of air which, in that particular case, was contaminated by the vapours of benzene, white spirit, and other solvents. If this method of applying paint is not liked in this country, much might be done by the opening of doors and windows of a newly painted room. The question of individual susceptibility has also to be considered, but one may say with a good deal of confidence that the prevention of dust, or its removal by fans or otherwise at the moment of its formation, and the removal of vapours or volatile solvents and the substitution of fresh air will bring cases of painters' colic, whether properly attributed to lead, to turpentine, or any other substance, down to insignificant proportions, and that no other remedy will be equally efficacious.

I am sorry to say that this sort of reasoning does not appeal to such a body as the International Labour Office, and that the very polyglot body which will in due course meet at Geneva will pay but little attention to medical evidence. The Treaty of Peace signed at Versailles provided for the holding of international conferences on questions in which labour is interested, such conferences to be attended by representatives of the governments, the employers, and the workers of every country, and each country is under an obligation to carry out so far as practicable the decisions arrived at by the conferences.

When I was at Washington rather more than a year ago at the first of these conferences we had to consider the health of the workers, and, in particular, the prevention of anthrax, of mercury poisoning, of phosphorus poisoning, and of lead poisoning in so far as it affected women and young persons. We made certain recommendations, one of which has in substance been embodied in the Women and Young Persons (Employment in Lead Processes) Bill, which became an Act of Parliament last session. I am bound to say that though I have in the past had a good deal to do with Acts of Parliament, I did not realise until I was at Washington the extraordinary difficulty of devising rules which would achieve the desired result without unnecessary dislocation of trade, and which were capable of being applied to a variety of countries with different industrial conditions and different methods of domestic legislation. With my recent experience of the Lead Processes Bill, I now realise the difficulties still more acutely, but even at Washington I thought it desirable to suggest to the conference that in view of the complexity of industrial diseases the problems attached to them should be carefully considered beforehand by a committee on which the governments, the employers, and the workers should all be represented. The Conference at Washington unanimously recommended the appointment of such a committee, but no such committee has been so far appointed, and a purely bureaucratic organisation is tackling the problems instead. Governments are particularly susceptible to public opinion, and the public opinion of to-day is largely based on the imperfect recollection of the cases of lead poisoning which occurred five-and-twenty years ago. An instance of this was brought to my notice during the passage of the above-mentioned Bill through

Parliament last session. In supporting the Bill one speaker gave an instance of lead poisoning which had occurred in the paint trade. He gave various details, and I had the incident he mentioned looked into and found it was a case of poisoning by arsenite of copper. This is typical of what happens. The conditions five-and-twenty years ago were so bad, and made such an impression on the social workers of the day, that many people believe that they prevail yet. The fact is that the Home Office, under the guidance of Dr. Legge, has very much reduced lead poisoning, and in the pottery industry has to all intents and purposes abolished it, for there are in that trade no new cases, and the cases which are from time to time reported in the china and earthenware statistics are the deaths of workers as they gradually get old, who contracted lead poisoning many years ago, before the Home Office regulations came into force. But though these facts are known to a few of us, the public as a whole, not only in this country, but in other countries, have not had time to bring their knowledge up to date, and, stimulated by their recollection of bygone days, they have brought pressure to bear on the various governments of the world, and the officials who represent them, to treat this problem as if it still was in the same state as it was a generation ago. The International Labour Office shows no inclination to have such problems scientifically examined by the employers and workers who are most nearly concerned, but they have appointed a permanent official, who seems to be entrusted with the duty of preparing a case for the prohibition of lead, and who has manifested no anxiety to have the assistance of such a committee as was recommended at Washington.

It is not very easy, but it is possible, to frame regulations which would reduce to a minimum the vapours and dust to which painters are exposed, and you may ask me why such regulations have not been, at any rate, given a trial. The answer is that in some foreign countries—for instance, Austria, Germany, and Belgium—they have been tried and have proved beneficial, and I am not without hope that the Home Government may give them a trial. Government Departments at the present time have their hands full of problems, and one cannot blame any department for not desiring to add more to its already heavy load. But I think the Home Office must pay the penalty of success. It has done so much to improve the condition of the workers with so little interference with trade; it has in the past co-operated fairly and reasonably with trades unions and manufacturers, and the tradition which it has created is one not to be lightly destroyed. We chemical manufacturers and chemists, recognising the importance of chemistry to the nation and the fact that we are now only in the beginning of a highly specialised chemical industry, must bring pressure to bear on the Home Office and all other Government Departments concerned to maintain the high standard they have themselves set up and not to be led away by the false gods of the Geneva bureaucracy. The International Labour Office may be productive of much good, but in so far as industrial hygiene is concerned it will accomplish nothing without the co-operation of those primarily affected, and its present methods are tending to promote suspicion rather than confidence.

#### References.

- <sup>1</sup> Oliver, "Dangerous Trades," p. 429.
- <sup>2</sup> Journal of State Medicine, Vol. XXVIII., p. 306.
- <sup>3</sup> Home Office Report for 1918, p. 79.
- <sup>4</sup> Legge and Goadby, p. 109.
- <sup>5</sup> Journal of Industrial Hygiene, Vol. II., p. 129.
- <sup>6</sup> See Journal of Industrial Hygiene, Vol. II., p. 72.
- <sup>7</sup> Home Office Report for 1918, p. 66.
- <sup>8</sup> Home Office Report for 1919, p. 61.
- <sup>9</sup> Legge and Goadby, p. 120.
- <sup>10</sup> Legge and Goadby, p. 66.

## BRITISH CHEMICAL PLANT MANUFACTURERS' ASSOCIATION.

Prior to the war British chemical manufacturers procured much of their plant from Germany owing to the difficulty they experienced in obtaining plant in this country which satisfied their requirements. A feeling had long prevailed in the industry that steps should be taken to promote closer co-operation between British chemical and British chemical-plant manufacturers. The need for such action was fully realised by those members of the Association of British Chemical Manufacturers who accompanied the mission sent out by that body under the aegis of the Department of Overseas Trade to inspect chemical factories in the Occupied Area of Germany in May, 1919.

A year later it was decided to invite representatives of the plant manufacturers to meet in conference representatives of the Fine Chemical Group of the Association of British Chemical Manufacturers, to whom the matter was of particular importance. The plant manufacturers agreed that it was desirable that they should be brought more closely into touch with the chemical manufacturers and kept more adequately informed of their requirements, and expressed their willingness to do all in their power to assist the chemical manufacturers. The two conferences then held led to the formation of the British Chemical Plant Manufacturers' Association, which at its inaugural meeting in July last consisted of nineteen firms. Mr. J. H. Rawson (The Widnes Foundry Co., Ltd.), Mr. L. M. G. Fraser (Messrs. W. J. Fraser and Co., Ltd.), and Mr. E. A. Allott (Messrs. Manlove Allott and Co., Ltd.) were elected chairman, vice-chairman, and hon. treasurer, respectively, and Mr. W. J. U. Woolcock (General Manager of the Association of British Chemical Manufacturers), secretary. The new Association became affiliated to the Association of British Chemical Manufacturers.

It was agreed that one of the first questions to be considered was the standardisation of chemical plant and apparatus, in which the Germans have reached a very advanced stage. At a joint meeting of representatives of the two Associations it was decided that the question of standardising jacketed-pans with stirrers and covers, boiling-pans and filter-press plant should be the first to receive consideration. The joint sub-committees which were set up to consider these matters are making good progress. Both the British Chemical Plant Manufacturers' Association and the Association of British Chemical Manufacturers are represented on the Sectional Committee on Chemical Engineering of the British Engineering Standards Association, which is also considering the standardisation of chemical plant and apparatus. A joint research committee was also appointed to investigate any new types of plant which may be reported to be in use in other countries and to go into any questions bearing upon the improvement of chemical plant in this country. This committee has invited the co-operation of the Chemical Engineering Group of the Society of Chemical Industry.

Attention having been drawn to the fact that the German plant manufacturers utilise nickel to a much greater extent than the British plant manufacturers, the Committee has now under consideration the uses to which this metal could be put in chemical plant. In this connexion very valuable assistance is being received from the Mond Nickel Co. The question of the use of 12 per cent. chromium steel is also being investigated.

The membership of the British Chemical Plant Manufacturers' Association has increased considerably since its inauguration, and it is hoped that, as the Association becomes more fully representative

of the various branches of the manufacture of chemical plant, the object for which is was formed will be achieved, namely, to ensure that British chemicals shall be made with British plant.

## THE HABER PROCESS AT MERSEBURG (SAXONY).

J. R. PARTINGTON.

The Haber process carried out at Merseburg differs from that at Oppau (cf. J., 1921, 99 **x**) in two respects:—(i) The mixture of nitrogen and hydrogen is made directly by treating a mixture of producer (air) gas and water gas, with the addition of steam, by the Bosch process (B.P. 14,508 of 1920); (ii) the purified mixture of nitrogen and hydrogen, before passing to the main catalyst furnaces, is passed through a series of small catalyst furnaces ("Vorofen") to remove impurities (B.P. 5335 of 1911).

There are 31 gas generators, 5 for air gas and the rest for water gas, all the air being provided by two blowers, each of 350 h.-p. The gases are passed through three gasholders, each of 1,765,000 cu. ft. capacity, providing a reserve for fifteen minutes. From these the gases are taken and mixed by blowers. The mixed gas then takes up the requisite amount of water vapour in two rows of six towers, each 82 ft. high, through which hot water is pumped by seven pumps, each of 70 h.-p. The water is warmed in a tower by the gases leaving the heat-exchanger of the contact furnaces for the hydrogen production. There are 48 heat-exchanger towers.

The gases, containing hydrogen, nitrogen, carbon monoxide and some carbon dioxide, now pass, after preheating, to two sets of 24 contact furnaces in which the reaction  $\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{CO}_2 + \text{H}_2$  occurs. The gas passes to two gasholders, one of 350,000 cu. ft. and the other of 1,050,000 cu. ft. capacity. Carbon dioxide is removed by washing in 21 to 30 towers at 25 atm., the compression of the gas being in large part effected by one of the cylinders of the 200 atm.-compressors, although a few pumps for 25 atm. are provided. Pelton wheels recover about 40 per cent. of the energy of the released gas, which is utilised to compress the water to 25 atm. The power for this part of the plant is 40,000 h.-p.

For the removal of the carbon monoxide, the gas compressed to 200 atm. by twenty-four 1000 h.-p. compressors and twelve 500 h.-p. compressors is scrubbed in 19 towers with ammoniacal copper formate solution circulated by nine double pumps of 600 h.-p. The carbon monoxide recovered by reducing the pressure in the towers is sent along with water to the hydrogen contact-plant. Six towers for caustic soda remove the remaining carbon monoxide, the soda being circulated by six small pumps.

The gas now passes to nine small contact furnaces ("Vorofen"), in which the remaining impurities are removed by catalyst mass before passing to the main contact furnaces. There are twenty-four main catalyst furnaces, five being in reserve, and twenty-four heat-exchangers. Circulation is effected by eleven pumps of 700 h.-p. each ("Umlauf-pumpen"). The ammonia formed is absorbed in water, compressed to 200 atm. by five 100 h.-p. and two 300 h.-p. pumps in twenty towers 39.3 ft. high and 29 ins. diameter. The ammonia solution is passed to an expander, the ammonia gas evolved being absorbed in a separate tower. In the expansion the hydrogen and nitrogen gases dissolved in the water under 200 atm. pressure are liberated; these pass through the ammonia scrubbing tower

and are collected in a gasholder, from which they re-enter the circulatory system.

The capacity of the Merseburg factory, when completed, will be 800 tons of ammonia per day.

## SOCIETY OF CHEMICAL INDUSTRY.

### APRIL MEETING OF COUNCIL.

Among the matters considered at the meeting held on April 15, Sir William J. Pope presiding, was a suggestion made by the Institution of Mechanical Engineers that a further joint meeting of the two societies might be held in the early part of next session. The Institution proposed that the paper should be contributed by one of its members, and should deal with some subject of common interest, such as the construction and manufacture of large-scale chemical plant. The suggestion was cordially approved. The proposal put forward by the Chemical Society is dealt with elsewhere in this issue (p. 139 **x**).

Sir William Pearce was re-appointed the Society's representative on the Governing Body of the Imperial College of Science and Technology for a period of four years from June 1 next; and the resignation from the Council of Mr. W. J. A. Butterfield on account of pressure of work was accepted with regret. It was reported that the following retire from the Council after the Annual General Meeting:—(a) Vice-presidents: Prof. W. R. Hodgkinson, Mr. Robert Mond, and Mr. W. F. Reid. Mr. S. R. Trotman also retires but is eligible for re-election. (b) Ordinary members: Mr. C. S. Garland, Sir R. Hadfield, and Dr. F. M. Perkin.

The Government and Parliamentary Committee reported that it had brought before the Board of Trade certain points in the Dangerous Drugs Act, 1920, which may prevent or restrict the development of scientific research in the particular chemicals to which the Act refers. The renewal of the Society's subscription to the Conjoint Board was authorised. Twenty-eight new members were elected; and the report of the Manchester Section was submitted and approved.

### ANNUAL MEETING, 1921.

#### MCGILL UNIVERSITY, MONTREAL.

It will be of particular interest to members who are going to Canada to attend the annual meeting of the Society in August, to know that many of the members they will meet there as manufacturers, or associated with manufacturers, are graduates of the university which owes its existence to the exceptional foresight of the Scotchman James McGill, who was born in Glasgow in October, 1744. Exactly one hundred years ago, in 1821, the charter for the establishment of McGill University was granted.

When the visiting members first see the University they will find it difficult to realise that the fine site the University buildings occupy in the heart of Montreal was wild pasture land less than a century ago. But the rapidity of development characteristic of Canada is exemplified in this seat of learning as well as in commerce, manufacturing, and agriculture, and an idea of the vast strides that have been made may be gathered from the fact that when James McGill was one of Montreal's leading merchants the town consisted of only 9000 people, whereas the city's population is now approaching one million. Prominent among the buildings is that of the medical faculty, which brings to mind some of the men most famous in



medical science. McGill University has given to the mother country two of her most illustrious professors, Sir W. Osler and Sir E. Rutherford.

Associated with the University are the Forest Products Laboratories of the Dominion Government, the function of which is research and technical service relating to woods and all products derived therefrom. The fact that Canada is the third largest timber-producing country in the world and the largest within the Empire indicates the importance of this work. Canada was the first country in the Empire to establish laboratories of this kind, but the value of research in connexion with forest products is now recognised, and similar institutions have been or are being established in India, in Australia, Great Britain, and Burma. The laboratories at present are organised into four research and technical service divisions, namely, pulp and paper, timber, physics, timber tests, and wood preservation. Visitors interested in work of this kind will be made welcome and conducted through the institution by a member of the staff.

Members and their friends who propose to attend the Annual Meeting in Montreal and/or the subsequent proceedings in Canada and the United States are reminded that they will be heartily welcome to travel with the President and his party by the s.s. "Megantic," which leaves Liverpool for Montreal on August 12; and that they should notify the General Secretary of their intention without delay.

#### NEWS FROM THE SECTIONS.

##### NOTTINGHAM.

At a meeting held on April 13, Mr. S. II. Burford gave an informal account of the work of the Public Analyst. The variety and amount of the information required concerning each sample have much increased in recent times. An analyst must be a student all his life; he must have a wide knowledge of the chemical and legal sides of his profession, and must be able to prepare reasoned and judicial reports. Mr. Burford suggested that the time was ripe for the institution of official laboratories throughout the country, staffed by full-time assistants, and each in charge of an expert who should deal with special substances, e.g., water and sewage in one laboratory, oils and fats in another.

The chairman, Mr. J. H. Dunford, noted that under the Fertilisers Act it might be an offence to sell a product having a higher percentage than that demanded by the Act. Mr. J. White considered that the cares and variety of the work lent it interest. Milk which was clean when first produced should be kept so, not contaminated and then purified. Major S. R. Trotman agreed as to the exacting work of the profession. The analyst had little or no time for research into analytical methods. Mr. H. Droop Richmond said that the analyst, as a consultant, was kept in touch with analytical problems, and he simply had to make time to investigate them. Mr. S. J. Pentecost thought that the results of the Public Analyst should be made available to the public as were those of Medical Officers of Health. Mr. J. M. Wilkie referred to a recent paper by Cofman on the French system of territorial laboratories; and expressed the opinion that the Public Analyst should be better remunerated so that he might have a proper staff. Mr. Burford, in reply, and also Mr. H. Droop Richmond, remarked upon the uniformity of milk samples at the present day, which they ascribed to the elimination of the small dealer, or the blending of small supplies, or the control by analysts employed by large firms.

#### MEETINGS OF OTHER SOCIETIES.

##### FARADAY AND OTHER SOCIETIES.

A general discussion, organised by the Faraday Society with the co-operation of several of the technical societies, was held on April 6 in the hall of the Institution of Mechanical Engineers, the subject being "The Failure of Metals under Internal and Prolonged Stress." As nearly all of the 17 papers presented had been printed and circulated beforehand, it was possible to confine the actual presentation of the papers to a few minutes, leaving for the discussions the greater part of the six hours occupied by the three sessions into which the meeting was divided. This arrangement was found to be very successful, and the subject was treated from many points of view, metallurgists, engineers, and pure physicists contributing their quota of information and opinion. Prof. A. W. Porter, Capt. H. Riall Sankey, and Sir George Goodwin, as presidents of three of the co-operating societies, presided in turn.

Dr. W. Rosenhain, in an introductory address, explained how the hypothesis of an inter-crystalline cement had been developed to account for the phenomena of the time-effect in the flow of metals under stress and the spontaneous cracking of cold-worked material. This hypothesis received strong support from Sir Alfred Ewing, speaking from the standpoint of the crystallographer, and it must be said that, although numerous objections to the hypothesis from the theoretical and practical sides were brought forward, no other hypothesis capable of explaining the whole of the facts was suggested. In the discussion of the general question much stress was laid on the part played by corrosion in the initiation of season-cracking, the important investigations of Mr. H. Moore and his colleagues in the Research Laboratory of Woolwich Arsenal containing evidence, derived from a large quantity of experimental material, that such cracking only occurs when chemical action has taken place. Some speakers considered that cracking also occurred in the absence of chemical action, but such a statement is naturally difficult to prove, since minute traces of ammonia, for instance, are sufficient to initiate the action. Prof. C. H. Desch reviewed the facts concerning the inter-crystalline nature of the attack of certain chemical reagents on various metals and alloys, and laid stress on the selective action in such cases. Evidence of the effect of time in bringing about fracture under comparatively low stresses was afforded by Mr. L. Archbutt's observations on the failure of the lead sheathing of telegraph cables, the metal showing inter-crystalline brittleness without chemical corrosion. Dr. W. H. Hatfield's paper opposed the hypothesis of an amorphous film, continuing a controversy begun at a recent meeting of the Institute of Metals.

After the general papers, two groups of papers dealing respectively with steel and with the brasses and similar alloys were taken. Sir Henry Fowler described the failure of some steel locomotive boiler tubes, and several speakers dealt with the remarkable brittleness in steel caused by the action of hot solutions of alkalis and certain salts. Cases of inter-crystalline failure in steel are comparatively rare, but have a special interest. The season-cracking of brass was dealt with from several aspects, much experience having been gained during the war in regard to the behaviour of brass tubes, cartridge cases, and other objects. There were several contributions from Woolwich Arsenal on this subject, and it was shown that the method recommended there and adopted by some manufacturers of annealing at a temperature too low to cause softening of the hard-worked metal



is an effectual preventive of season-cracking. It was stated, however, by some speakers that when a correct draft is taken in the drawing of tubes, for instance, the setting up of severe internal stresses is avoided, and annealing becomes unnecessary. The almost explosive cracking of hardened steels was discussed, a paper on this subject by Mr. J. N. Greenwood entering deeply into the theory as well as the practical experience in hardening shops.

The joint discussion must be considered as one of the most valuable of those which have been held under the auspices of the Faraday Society, and the interchange of views has done much to prepare the way for a comprehensive survey of the whole subject of failure under stresses of the kind which present themselves in season-cracking, slowly developing brittleness, and other forms of intercrystalline weakness.

#### NATIONAL UNION OF SCIENTIFIC WORKERS.

The half-yearly meeting of the Council was held on April 9, in London, the president, Prof. L. Bairstow, presiding. Among the resolutions passed unanimously were the following:—

"That this Council views with misgiving the subordination of scientific workers controlling scientific staffs to non-scientific officials in Government departments. It considers that it is in the best interests of the State that its scientific officers should enjoy the greatest possible freedom from lay interference in connexion with their duties, and should be granted at least equal status and remuneration to that of officials of the highest class in the Civil Service."

"That this Council, having regard to the existing demand for increased educational facilities, deprecates the growing tendency of public bodies to reduce expenditure on education, particularly in neglecting to provide for further institutions for the study of science and technology, and by threatening existing institutions with closure, irrespective of their national utility."

A third resolution deprecated the tendency to discriminate between the sexes in the payment of salaries of scientific workers; a fourth urged freedom in research and the undesirability of research work demanding privacy in its pursuit and restriction in publication being undertaken under the auspices of a university; and other resolutions were carried approving the appointment of a consultative committee of representatives of Government branches, and of allowing reduced subscriptions to members resident abroad and to research students and apprentices receiving small grants or salaries.

On April 14 Mr. H. E. Potts, of Liverpool, addressed the London Branch on "The Position of Employer and Scientific Worker in relation to Patent Law." Prof. L. Bairstow presided.

Mr. Potts dealt with the present legal position, the basic principle of which is that in the absence of special contract the invention of an employee, even though made in the time, with the materials, and at the expense of the employer, does not become the property of the employer so as to prevent the employee from taking out a patent. There are, however, many exceptions, and the higher the position of the employee the more likely they are to apply. The four principal exceptions are:—(1) The work may be done under specific instructions, in which case the director may claim to be the inventor and his claim allowed. (2) The work may be considered as a condition of employment and to be included in the duties of the position held. (3) The worker may be in a position of special confidence and have access to special information. The case quoted was that of an English agent for an

American firm who attempted to patent in England an improvement on the firm's design. (4) There is frequently an agreement in force between firms and their scientific staffs that all patents arising out of work done shall be assigned to the firm. In return some firms give bonuses for successful inventions, others a share of the proceeds or royalties, and it sometimes happens that a firm only claims a free licence and leaves all royalties to the inventor. Mr. Potts is of the opinion that the Union should decide what is a fair agreement, and that some form of bonus for successful inventions should be offered to stimulate effort in the final stages of invention. He suggested that before formulating a policy in regard to inventions of scientific workers in industry, the Union should investigate the conditions prevailing in America and on the Continent, and should confer with some representative body of employers.

#### ROYAL PHOTOGRAPHIC SOCIETY.

The meeting held on April 12, arranged by the Scientific and Technical Group, was devoted to a consideration of some aspects of cinematography. Mr. R. J. Trump described an ingenious device for a shutterless, continuous-feed, projecting cinematograph. The light from the projecting lens is reflected by a plane stationary mirror on to the inner periphery of a large wheel rotating synchronously with the movement of the film. The wheel carries a number of plane mirrors arranged in sets of three and disposed at such angles that the image of the moving picture is kept stationary on the screen. An absence of flicker and a reduction of wear and tear of film and mechanism are the advantages claimed for the system.

Mr. H. M. Lomas dealt generally with the limitations imposed by the materials at present available for cinematographic purposes and the difficulties arising from them. Amongst the things specifically mentioned were "static markings," marks produced in the unwinding of the film caused by light flashes due to electrification, and the coarseness of grain of the emulsion which limits definitely the amount of magnification allowable.

#### THE PHYSICAL SOCIETY.

At the meeting held on April 8, Sir W. H. Bragg read a paper on "The Application of the Ionisation Spectrometer to the Determination of the Structure of Minute Crystals." The method of X-ray analysis of the structure of minute crystals as developed by Debye and Hull necessitates the use of heavy discharges in the X-ray bulb and long exposure of the photographic plate. In the present method, the crystal powder is pasted on a flat surface and placed on the spectrometer table in the manner employed for a large single crystal. The intensity of the diffracted X-ray spectrum corresponding to various angles is measured in the ionisation chamber of the spectrometer in the usual manner. A bulb current of one milliamperè is sufficient, and the main part of the spectrum can be determined in about half an hour. The paper was illustrated by the spectra of aluminium, silicon, and lithium fluoride determined in this manner.

A paper on "A New Registering Microphotometer" was submitted by Dr. W. J. H. Moll, of Utrecht. The instrument is designed to record the respective densities of very small areas of a photographic plate. A small incandescent lamp is fitted with a condenser, whereby an image of the glowing filament is cast upon a slit. The photographic plate is disposed between two microscope objectives in such manner that an enlarged image of the illuminated portion of the plate is produced at a second slit through which radiation transmitted by the

plate passes and falls upon an extremely sensitive and robust type of thermopile designed by the author. The plate can be given a slow motion by a micrometer screw, and the beam of light reflected from the mirror of the galvanometer connected with the thermopile records the intensity of the radiation transmitted by the illumined portion of the plate. The device is readily applicable to the investigation of the density of photographic negatives of all kinds, and the extremely sensitive thermopile employed should find application in a large number of other directions.

#### ROYAL SOCIETY OF ARTS.

A paper on "Low Temperature Carbonisation and Smokeless Fuel" was read by Prof. H. E. Armstrong on April 13, Sir Arthur Duckham presiding.

In the first part of his paper, Prof. Armstrong dealt with the use and abuse of coal as fuel, and outlined the early history of "coalite." Early in 1913 a large German company was about to associate itself with the English undertaking, but the war intervened, and it was not until 1919 that development work was resumed at Barnsley. A carbonising plant built to the design of Mr. T. M. Davidson had been in operation since November, 1920, and was undoubtedly a success. The trouble due to expansion of the fuel on carbonisation had been overcome by building the ovens with upright walls and fitting them with perforated hanging plates movable in a vertical plane over a narrow range; the coal was filled into the space between the side of the oven and the opposed face of each plate, thus enabling the width to be varied. During carbonisation the plates exerted pressure on the coal, and the volatile matter escaped through the perforations. When carbonisation was complete, after 5—8 hours, the "coalite" slabs were allowed to fall into an air-tight, water-cooled chamber. It was essential that the "coalite" should not be exposed to air during cooling. The product contained 10—15 per cent. of volatile matter and burned with a bright flame; the coal oil was well within the Admiralty specification for fuel oils; and motor spirit could be obtained from the liquid and scrubbed from the gas.

In the long discussion which followed, the chairman said that he had seen the plant at Barnsley and was convinced of its success. The great drawback to the process, however, was the lack of an absolutely satisfactory balance-sheet, and this was the reason why gas engineers had fought shy of it; the success of the process would depend on the value of the "coalite" and the value of the oil. Dr. J. A. Harker said that in an investigation carried out under the auspices of the Ministry of Munitions, it was ascertained that from 1 ton of coal the yield of crude "coalite" was 14 cwt., of which rather less than 13 cwt. was saleable. Economically, the process depended upon selling the 13 cwt. for the same price as that paid for 1 ton of the coal, and of paying operating costs from the sale of by-products. He thought that the margin between profit and loss would be very narrow. Among the points raised by subsequent speakers were the friability of fuel produced by low-temperature carbonisation processes and the necessity for briquetting, the economy that should be obtainable with the Neilsen process of internal-heating of the retorts with water gas, and the need of eliminating as much ash as possible by properly washing the coal.

In his reply, Prof. Armstrong said that, as a professor, he was not concerned with the cost of the process. The public should be prepared to pay more for "coalite" than for coal because it did not burn

away so rapidly and was smokeless. The yield of fuel oil was about 1 ton from 10 tons of coal.

#### THE CHEMICAL SOCIETY.

A meeting was held at the Institution of Mechanical Engineers on April 7, Sir William Pope presiding, when Dr. F. W. Aston delivered a lecture on "Mass Spectra and Atomic Weights." On April 21 an ordinary scientific meeting was held at Burlington House. Sir James Walker, the new president, gave a short address, and then six of the nine papers on the agenda were presented in abstract. The paper of most immediate interest to technical chemists was that by Dr. J. A. Newton Friend on "A Colloid Theory of the Corrosion and Passivity of Iron and of Ferrous Salts," of which the object was to show that the reactions involved are not purely ionic but colloidal.

When iron is exposed to moving water containing dissolved air the rate of corrosion increases rapidly with the flow of the water until it reaches a maximum, after which further increase in the velocity of the water causes a retardation of corrosion, none taking place at about five miles per hour. It was suggested that iron slowly oxidises in aerated water to form colloidal ferrous hydroxide, which then undergoes alternate oxidation by air and reduction in contact with metallic iron, the latter suffering rapid corrosion. If, however, the colloid is swept from the surface of the iron as rapidly as it is formed this autocatalytic action cannot take place, and the iron oxidises but slowly. The theory was tested by determining whether or not the rate of corrosion is influenced by factors that increase the stability or effect the precipitation of positive sols. This was shown to be the case. Thus the presence of anions such as  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ , and  $\text{Cr}_2\text{O}_7^{2-}$  is known to cause the precipitation of colloidal iron hydroxide,  $\text{Cl}^-$  being least and  $\text{Cr}_2\text{O}_7^{2-}$  most effective. These ions also influence the rate of corrosion,  $\text{Cr}_2\text{O}_7^{2-}$  exerting the most marked inhibiting action and  $\text{Cl}^-$  the least. It was also shown that protective colloids, such as gelatin, acacia, and albumin, inhibit corrosion, and that there was a remarkably close relationship between their action in this respect and their influence on the catalytic activity of colloidal platinum upon hydrogen peroxide. Suspended colloids are very sensitive to "poisons," and attention was drawn to the fact that poisons also inhibit corrosion, arsenious oxide being particularly effective. Radium rays, which precipitate iron hydroxide sol, likewise retard corrosion. Colloidal iron hydroxide is very stable in pure water, and is not precipitated by boiling. Addition of small quantities of an electrolyte, such as sodium chloride, causes marked precipitation if the temperature is raised. An explanation is thus forthcoming of the remarkable and otherwise inexplicable fact that whereas sea-water is more corrosive than fresh water at  $10^\circ\text{C}$ , it is less corrosive at  $30^\circ\text{C}$ . It was pointed out that passivating agents are those which possess anions capable of precipitating positive sols; and the oxidation of ferrous sulphate in neutral or alkaline solution was attributed to the presence of a sol.

Dr. F. Challenger contributed a paper on the interaction of the halogen derivatives of tertiary aromatic bismuthines with organo-derivatives of magnesium and mercury; Prof. G. T. Morgan outlined some new work by himself and Mr. J. D. Smith on the constitution of simple and complex cobaltic quinonoxime lakes; and Dr. N. V. Sidgwick and collaborators presented three papers dealing with the influence of position on the solubility of the substituted benzoic acids, on the solubility and volatility of the mono- and dinitrophenols, and on the solubility and volatility of the chloro- and nitro-anilines and their acetyl derivatives.

## NEWS AND NOTES.

## AUSTRALIA.

**Cobalt Ore in Queensland.**—The Imperial Mineral Resources Bureau has received from the Department of Mines, Brisbane, an account of the development work which has been carried out on the recently discovered deposit of cobalt ore in the Cloncurry district (*cf.* J., 1920, 378 a). Two shafts, 250 ft. apart, have been sunk on the general strike (underlie), the first to a depth of 112 ft. and the second to 92 ft. In No. 1 shaft from the surface to 42 ft. the ore (cobaltite) averaged 4–5 per cent. metallic cobalt over an average width of 5 ft. A similar ore body was found at 80 ft. No. 2 shaft has met with ore varying from 1 to 3 ft. wide of similar value to that encountered in No. 1 shaft. Development work is not sufficiently advanced to show if the ore occurs in short shoots or as a continuous run.

**Shale Oil in New South Wales.**—The Joadja oil-shale mine, which was formerly worked by the Australian Kerosene Oil and Mineral Co. but closed down about 20 years ago as a result of American competition in oil, is now being reopened by the Shale Petrol Oil Co., Ltd., a new company that proposes to work the coal, which occurs abundantly on its property, as well as the shale. The mine is situated near Mittagong, on the main line between Sydney and Melbourne. On distillation, the shale gives 73 per cent. of volatile hydrocarbons and an average of 106 galls. of crude oil per ton of shale. The old company produced considerable quantities of motor spirit, which in those days was considered a nuisance and therefore burnt on the spot.

## Tasmania.

**Shale Oil Distillation.**—The Rialton-Latrobe shale works has started to send crude oil to Melbourne to be refined. The company owns shale deposits which are expected to produce about 300 million galls. of oil, and is installing a plant which should be able to distil about 350 tons of shale per day. Demand for oil is growing almost daily, owing to the steady increase in the use of motors for farming operations.

**Cement Manufacture.**—The hydro-electric plants which are being laid down in Tasmania are making very heavy demands upon the supplies of cement available in the island. As a result of this, new cement works are being erected by the National Portland Cement Co. on Maria Island, and by the Fingal Cement Co. on the mainland; the former company is planning an output of 30,000 tons per annum, and the latter is expecting to produce in July next.

**Lessons in Economic Independence.**—The manufacture of calcium carbide was developed in Tasmania during the war, and was safeguarded owing to shipping difficulties. It was just beginning to become well established when the shipping situation improved, and the old importers began to attempt to recover their market by offering carbide at a "cut" price. In consequence, the Federal Government prohibited the importation of carbide, except under licence, for a period of twelve months, by which time it is anticipated that the new tariff law will be in operation.

A different story is that of imported petrol, which is not produced in the country. The importers of this commodity (the price of which is fixed by law) applied to the Court for permission to increase the price, and when they were asked by the President of the Court what would happen if their request were refused, they replied that the importation would cease. The application was granted.

## JAPAN.

**The Camphor Industry.**—The efforts made by the Japanese Camphor Monopoly to increase production resulted in 1919 in a yield of nearly 6·7 million lb., of which 5·7 million lb. came from Formosa. Although this was nearly double the 1918 output, it was approximately 20 per cent. less than that for 1916, a year of record production. On account of the rapid growth of the Japanese celluloid industry, the exportable surplus is becoming smaller, and in 1919 the United States received only 1·42 million lb., compared with 2·5 million lb. before the war.—(*Chem. Ind., Mar. 14, 1921.*)

## FRANCE.

**Industrial Notes.**—*Coal.*—One immediate effect of the coal strike in Great Britain was to stimulate the French coal trade. The fear of a short supply of coal has been strongest in the south-west and south, where British coal is mainly consumed. It is estimated that the increase in orders arising from this cause is about 350,000 tons.

According to the report of the Central Committee of the French Coalfields, the production of coal in France rose from 22,341,864 metric tons in 1919 to 25,274,294 t. in 1920. Importations in 1920 were 24,056,262 t. of coal, 3,985,213 t. of coke, and 2,070,893 t. of briquettes, compared with 22,005,000 t., 1,723,619 t., and 1,180,139 t. respectively in 1919. British exports to France in 1920 amounted to 11,432,000 t. of coal, 687,000 t. of coke, and 887,000 t. of briquettes, and showed a great reduction over the figures for 1919; this was due to increased supplies from Germany and the Saar basin, and to importations from the United States. The total supplies in the past year were thus 48,359,000 t. of coal, 4,767,000 t. of coke, and 4,129,000 t. of briquettes, or 4 million tons of coal and 2½ million tons of coke in excess of the supplies in 1919; but the ratio of production to consumption, 50 per cent., compares unfavourably with that for the last pre-war year, viz., 65 per cent. The production of the Saur and Lorraine coalfields—about 7 million tons—is about 4 million tons less than the consumption in the recovered provinces. There is, moreover, little prospect of increased output in view of the 8-hour day and the diminished efficiency of the individual miner.

**Metallurgy.**—The greatly increased production of cast iron and steel resulting from the return of Alsace-Lorraine has emphasised the importance of finding outlets abroad, and this question is being considered by the Ministries of Commerce, Public Works, and Industrial Reconstruction in conjunction with representatives of the industry. As a result of combination between important metallurgical firms, production costs have been reduced and the general commercial organisation improved and strengthened so as to obtain a firmer hold on foreign markets. The Comptoir Siderurgique has lately added to its list of members the iron and steel firms of the Saar basin, but as these refuse to enter the export trade, the Comptoir is at present dealing solely with the home trade, although its chief function should be to promote exportation.

**Chemical Industry.**—Soap can now be exported freely, and it is to be hoped that complete freedom will soon be forthcoming, as this would be followed by a material reduction in prices. The slump on the chemical market still continues, and were it not for exportation the situation would be critical. Abroad there is acute competition from England, Germany, and the United States, and the last-named country is daily gaining a firmer hold on the South American market, formerly supplied by Germany.

The projected publication of all chemical abstracts in *Chimie et Industrie* and *Le Bulletin de*

la Société chimique de France (cf. J., 1921, 127 n) is generally welcomed by chemists, if only on account of the time that will be saved in searching in one or two journals for matter which may now be dispersed in many.

#### UNITED STATES.

**Recent Research in Applied Chemistry.—Carbon Black.**—A new process for producing carbon black is being rapidly perfected. It consists in forcing natural gas, consisting of nearly pure methane, through molten metal and collecting the resulting carbon black by the Cottrell process, hydrogen being liberated as a by-product.

**Syrup from Sweet Potatoes.**—The Bureau of Chemistry has devised a process of manufacturing a table syrup from sweet potatoes, of which large quantities do not now reach the market. The syrup is rich in sugar, has a good brown colour, is palatable, and suitable for baking, for making sweets, and for table purposes. It is considered by many to be equal to a high-grade, cane-sugar table syrup.

**Tarnishing of Metal Surfaces.**—A research laboratory which is investigating problems relating to the protection of metal surfaces has found that tarnish on highly-polished machine parts is often due to sunlight. This result is brought about by very slow breaking down of some of the hydrocarbons in the lubricating oil, carbon being deposited and hydrogen set free. The brown stain produced is indelible, but harmless.

**Testing the Fastness to Light of Dyes.**—A series of tests to ascertain the best means of determining the fastness to light of dyes has been completed, and the decision reached that the violet carbon arc gives results which agree best with the effect of sunlight. The white-flame carbon arc gave rather better results, but was only half as rapid. These carbon arcs were found to be superior in constancy to the mercury arc.

**Chlorination of Natural Gas.**—The chlorination of natural gas in localities where other economical use is excluded seems a certainty for the near future. Investigations conducted by the Bureau of Mines have shown the possibility of producing the four chloromethanes, etc., with a 90 per cent. or better yield, and the only remaining problem is that of heat control.

**Applications of Carbonyl Chloride.**—Phosgene is known to be very reactive at or above 450° C., and Dr. Charles Baskerville has utilised it to convert the oxides of aluminium and cerium, insoluble in acids, and the oxides of zirconium and thorium, which are soluble only in boiling concentrated sulphuric acid, directly into water-soluble chlorides or oxychlorides. Bauxite and carborundum gave ferric and aluminium chlorides, and zircon yielded zirconium chloride, the ferric chloride from the contaminating iron being distilled off owing to its greater volatility. The method consists in simply beating the pulverised material in a quartz tube in a current of the gas.

**Wood-alcohol Poisoning.**—Many deaths have occurred in the United States as the result of drinking wood alcohol, and various preventive measures have been discussed. It was pointed out by Mr. A. E. Outerbridge that the addition of a small amount of fluorescein to denatured alcohol, although invisible in the concentrated solution, would give on dilution a yellowish-green fluorescence that would immediately be noticeable and would therefore serve as a deterrent. Accordingly a sample of denatured alcohol containing 7 mg. of fluorescein per 100 c.c. was submitted to the Prohibition Commissioner, who gave the opinion, in a letter to *Chemical and Metallurgical Engineering* of March 2, that it was inexpedient to use fluorescein for this purpose, as the colour that becomes

apparent on dilution might prove objectionable in some manufacturing processes; the voluntary addition of fluorescein to a factory stock of completely or specially denatured alcohol, however, was highly desirable.

**Potash Discovery in Texas.**—A large deposit of alunite has been discovered near Floresville, Texas. Three outcrops of the mineral occur, separated from one another by six to twelve miles, and the outcrop of highest grade mineral covers about 20 acres. The commercial value of the alunite is said to be about \$250 per ton, and should it be possible to exploit the deposits commercially, an important source of potash will be made available. (*Oil, Paint and Drug Rep.*, Mar. 14, 1921.)

**The American Oil Chemists' Society.**—For many years the leading chemists in the cottonseed oil industry have been associated in the Society of Cotton Products Analysts, an organisation which was devoted primarily to perfecting methods of analysis used in the cottonseed industry. Certain specific qualifications for membership limited this society in numbers to those highly specialised but ensured a large amount of important investigation work. During the war the United States was drawn into a large international trade in several of the vegetable oils which became keen competitors of cotton oil, and this caused a commensurate widening of the society's interests with the result that at its annual meeting in May, 1920, it was re-organised under the name of the American Oil Chemists' Society. All persons engaged in chemical work relating to fats, oils, waxes, and allied substances are eligible for active membership in the new society, provided they have had at least five years' chemical training. Meetings are held once a year, usually in May, and in conjunction with the Inter-State Cotton Crushers' Association. The Society publishes regularly its transactions and maintains the Chemists Section of the Cotton Oil Press, a journal devoted exclusively to the interests of the edible vegetable-oil industry. Membership, however, is not limited to chemists and technologists engaged exclusively in the edible oil trade, but includes also those concerned with its industrial or technical aspects. Anyone interested in the activities of this new organisation may obtain further information from the secretary and treasurer, Mr. T. B. Caldwell, Wilmington, N.C.

#### BRITISH INDIA.

**Chemical and Allied Industries in Madras.**—The Acting Director of Industries, Madras, in a summary of industrial intelligence appearing in the first number of the *Journal of Indian Industries and Labour*, points out that the high price and scarcity of fuel is one of the main difficulties which hinder the development of industries in Southern India. Numerous discoveries of coal have been made in the Presidency from time to time, the most promising being those made in the Godavari and Kistna Districts. Endeavours to interest firms and syndicates in the exploitation of the fields have in so far been successful that a leading Madras firm has been granted a prospecting licence over a total area of 173 sq. miles in the Kistna district and over 80 sq. miles in the Godavari district.

The question of a possible cement industry at Bezvada has been investigated for the past three years, and it now seems probable that it will soon be established, as the Government has recently approved of the grant to a syndicate of rights in certain districts for mining and quarrying the limestone and shale required in the manufacture. One of the conditions governing the grant of the concession is that within four years an output of at least 30,000 tons of cement per annum must have been attained.

A collection of samples of aromatic gums and oleo-resins, available in commercial quantities in Madras forests, was made for a large London firm of manufacturing chemists, which was also supplied with full information about the sources of supply and quantities available. The firm is considering the establishment in Southern India of a factory for the manufacture of drugs and essential oils. Another London firm of manufacturing chemists is contemplating the erection at Vizagapatam of a factory for producing crude alkaloids, principally strychnine, from nux vomica. The firm has already acquired the necessary land, and a factory will be erected at an early date.

The exploitation and development of the deposits of phosphatic nodules in the Trichinopoly District, with a view to supplying this fertiliser at cheap rates to the ryots of the delta districts, has been under consideration for some time past. It is proposed to establish an experimental works for the production of finely-ground phosphate and for the exploitation of the gypsum and red ochre associated with the nodules.

The industrial chemist attached to the Department of Industries is investigating at the Indian Institute of Science, Bangalore, the fermentation vat for indigo dyeing, and the fermentation of prickly pear. The production of power alcohol and the suitability of South Indian woods for the manufacture of paper and pulp are also being considered.

The manufacture of inks and vinegar is being carried out at the Government Industrial Laboratory, Coonoor. Experiments in progress at the laboratory include the manufacture of casein glue, lacquer for tins, sealing wax, and an investigation into the available sources of pectin.

As a result of the experiments in the manufacture of glue which were undertaken by the Department, agreements have been made for the establishment of a small experimental factory in Madras. Other schemes being considered by private firms, with the assistance of the Department, are the manufacture of finished rubber goods, extraction of oil from groundnut and gingelly seeds, manufacture of pottery and porcelain ware, and a factory for the production of cane sugar.

**Second Forecast of Winter Oilseeds.**—The total area under rape and mustard, so far reported, amounts to 2,793,000 acres, or 22 per cent. below the revised figure at the corresponding date of last year. The total area under linseed is reported to be 1,784,000 acres, which is 23 per cent. below last year's second forecast. In both cases these figures exclude the "mixed" crop of the United Provinces. The condition of the crops is reported to be generally fair. —(*Ind. Tr. J.*, Mar. 18, 1921.)

#### SOUTH AFRICA.

##### Mining in the Protectorate of South-West Africa.

Since the country was opened for prospecting on March 1, 1920, there has been considerable mining activity; copper ore has been found in the Kaokoveld, galena and silver in the Kalkfontein district, discoveries of tin and iron are reported from the Usakos district, and oil-shales have been discovered in the Gibeon district. Copper ore traffic, which recommenced in August, 1919, has been steadily maintained, and an average of about 3000 t. per month has been dispatched during the last few months. The tin mines have been more active, and although they are not able to record a large production, owing to lack of machinery, the output has increased considerably. About 50 tons of scheelite was produced at the Natas mine in 1919, the output of marble has notably increased, and 948 tons of salt was dispatched by rail during the year. The value of the diamond output for 1919 was £1,500,000, the production 428,180

carats) showing a large increase over the previous year.

A writer in the *Star* (Johannesburg) states that the tinfields situated in the Karibib and Omaru districts extend over several hundreds of square miles and that their production will expand considerably. One of the properties has recently been acquired by a leading Rand mining house, and another has been taken up by an East London company. Mining methods have hitherto been very primitive, but modest profits have been won. The obstacles to development are lack of water and transport. —(*S. Afr. J. Ind.*, Feb., 1921.)

#### GENERAL.

**Finsbury Technical College.**—When the projected closing of the City and Guilds Technical College, Finsbury, was announced last July, students past and present, and many friends who appreciated the educational facilities offered at this College during nearly forty years, endeavoured to avert the disaster. A Defence Committee was formed which was composed largely of former students, and urgent protests were evoked almost spontaneously from the great professional institutions. As a result, two petitions were sent to the City and Guilds of London Institute, one signed by the presidents of many of the professional institutions, and the other signed by a large number of former students of the College and friends interested in its continuance. In reply the petitioners were informed that there was every hope that the College would be continued, with the help of the Board of Education and the London County Council, for a period of five years. The success of these negotiations has now been announced, and it is hoped that the future of the College is now assured. The members of the Defence Committee are very grateful for the help given by many friends of the College during the critical period which has now passed.

**The Imperial Mineral Resources Bureau.**—The second annual report, covering the year 1920, shows that excellent progress has been made in the organisation and work of the Bureau. Good headway has been made in the collection of information, both from the appointed corresponding members and from unofficial sources, and in its co-ordination and tabulation. The information so obtained is made available to all Government Departments both at home and overseas, as well as to co-operating technical institutions and societies; individuals and companies may also avail themselves of the service of the Bureau, but these are charged a nominal fee if information is asked for which involves special work by the staff. Important work is being done with a view to introducing a unified system of mining and metallurgical statistics, including the standardisation of weights and measures, and at the suggestion of the Bureau a resolution was passed at the Imperial Statistical Conference to the effect that when the projected Central Bureau of Statistics for the Empire comes into being, it shall work hand-in-hand with the Imperial Mineral Resources Bureau. It has also been arranged that the Bureau shall have charge of the mineral exhibit at the British Empire Exhibition to be held in 1923. The Technical Advisory Committees constitute an important feature of the organisation; they are fifteen in number and comprise 148 of the leading authorities on the respective minerals and the industries associated therewith. Sir Herbert Jackson is chairman of the Committee for Chemical Industries.

The work of indexing technical literature of the Empire and of foreign countries is being actively prosecuted, and during the year ten pamphlets, each relating to one metal or mineral, were published; twelve more were nearly ready for press

at the end of the year, and another eight were in course of preparation. Future publications of this type will include statistics relating to labour, health, and accidents. A special and comprehensive work on the "Iron Ore Resources of the World" is also being prepared. The Legal Section of the Bureau has completed two volumes dealing with Mining Law in Nigeria and West Africa, respectively, and a third on South Africa, with special reference to the Transvaal, is nearing completion. The Bureau was supported financially by grants which amounted to £20,452, of which £10,000 was from the Imperial Government, and the rest from the Overseas Dominions and the National Federation of Iron and Steel Manufacturers. The paid staff numbers 45 persons, exclusive of the chairman, Sir Richard Redmayne, and the vice-chairman.

"Cobalt (1913-1919)" (Imperial Mineral Resources Bureau pamphlet, 23 pp., price 9d.).—Although the results of research indicate the possible value of cobalt metal as a constituent of alloys and in electro-plating, commercial interest lies chiefly in the use of its salts in the pottery and enamelling trades. The oxide has largely superseded smalt (silicate) as a colouring agent owing to its greater purity, uniformity, and lower cost. For special purposes the carbonate, chloride, sulphate, and, less commonly, aluminate, phosphate, arsenate, and nitrate are also employed. As driers in the paint and varnish trade the acetate, resinate, and oleate of cobalt are used. During the war a cobalt-chromium alloy ("Stellite") found some application in the manufacture of cutting and milling tools.

The world's annual production of cobalt oxide up to 1913 amounted approximately to 250 tons. Before the discovery of cobalt ore in Ontario, New Caledonia produced over 80 per cent. of the world's supply. In recent years Canada has been the chief producing country, the actual figures for 1918, quoted from the Report of the Canadian Mines Department, being metallic cobalt 196 tons, cobalt oxide 512 tons, and other compounds 83 tons. The production of Katanga, in the Belgian Congo, promises to be of some importance, and considerable interest has lately been shown in a new deposit which is being actively exploited in the Cloncurry district of Queensland (*cf. J.*, 1920, 378 n, and this issue, p. 143 n).

The price of cobalt oxide in July, 1919, was quoted in London at 7s. 8d. per lb. For some unexplained reason it rose during 1920 to 20s. per lb., but has since fallen to 16s. per lb. nominal.

**British Yeast.**—The "Report on Yeast" by the sub-committee of the Standing Committee on Trusts (Cmd. 1216. 2d.) states that compressed distillers' yeast has almost entirely superseded brewers' yeast for use in bread-making, and that nearly the whole home-supply is produced by three firms, two of which are located in Edinburgh and one in Belfast. The pre-war selling price, inclusive of delivery, was about 19s. per 56-lb. basket, and the present price of about 39s. represents an increase in the cost of bread of less than 0.1d. per loaf. No information concerning production costs was forthcoming. From the time it leaves the distillery until it reaches the consumer, the yeast is controlled by the Wholesale Yeast Merchants' Association, together with its subsidiary yeast dealers' associations, and these in turn are controlled or influenced by the representatives or agents of the distillers; no person outside these associations could obtain supplies of British yeast.

Before the manufacture of compressed distillers' yeast was started in this country in 1886, foreign yeast was imported at the rate of about 282 tons per week, but owing to the competition of the home-produced material the imports had declined to about 200 t. per week immediately before the war.

From 1915 to 1917 imports were gradually restricted, and from April, 1917, to April, 1919, were prohibited. Importation was resumed in 1919, and now about 70 tons is being imported weekly. During the period of prohibition the output of British distilleries rose from about 400 t. to 650-700 t. a week, and at the present time the home consumption is estimated at about 635 t. a week. When, during the war, the distillers were invited by the Ministry of Munitions to enlarge their plants in order to meet the entire home demand, the Ministry is said to have given them some sort of assurance that importation would be restricted after the war; but this has been denied. The conclusion arrived at by the sub-committee is that unrestricted imports form the only safeguard against a combination of distillers arbitrarily fixing the price to the consumer, and should it be found necessary to protect the British distillers against imported yeast, such action should be preceded by legislation to protect the consumer by providing for the surveillance of trusts and monopolies.

**Use of Peat Fuel on Swedish Railways.**—Extensive trials have shown that peat, reduced to powder or briquetted, makes an excellent substitute for coal as fuel for locomotives (*cf. J.*, 1919, 145 n), and the Swedish Railway Board has taken over the plant at Hästagen bog, near Vislanda, which cost 975,000 kronor (krona=1s. 1½d. at par) and has a capacity of 30,000 tons per annum. The power required by the plant is small, and the peat is air-dried as far as possible, the hardest and driest lumps being used as ordinary fuel and the remainder pulverised or briquetted. Various new machines and devices for digging, transporting and drying peat have been devised by Engineer Eklund, and it is proposed to manufacture them in a new factory.—(*U.S. Com. Rep.*, Feb. 25, 1921.)

**The Norwegian Paper Industry.**—In this industry there are 26 cellulose mills, 63 wood-pulp grinding mills, 48 paper and cardboard factories, and about 15,000 workers. The production in 1918 was 438,438 tons of cellulose and wood pulp, and 116,889 t. of paper and cardboard. At present there is little demand for paper, stocks are accumulating, and the situation is not bright. Many mills have had to reduce their output, some have been closed, and about one-half of the 80 paper machines is lying idle. The production of wood pulp has as yet only been reduced where it is required for paper-making; stocks of cellulose and wood pulp are not so large as those of paper.—(*U.S. Com. Rep.*, Feb. 6, 1921.)

**Projected Salt and Magnesium Industries in Norway.**—The A.-S. Norske Saltreker is erecting a factory for the extraction of salt from sea water in Fjotlandsvaag. The factory will have a capacity of 5000 t. per annum, and will probably be finished in the summer. It is intended to utilise the magnesium salts for the production of about 100 tons of magnesium yearly, i.e., about one-quarter of the world's present production. The magnesium will in part be made into alloys for constructing motors, aircraft, balances, conductivity wiring, etc. For such purposes the alloy or metal will be covered with a protective film of oxide by a process, discovered by the engineer Backer, which consists in boiling in water under high pressure for about 15 minutes; the resulting coating is said to be firmly adherent even when hammered, and to act as an electrical insulator.—(*Chem. Ind.*, Mar. 23, 1921.)

**Belgian Coal Output in 1920.**—The Belgian output of coal in 1920 was 22,414,000 metric tons, which compares with 18,483,000 t. in 1919 and 22,842,000 t. in 1913. The average output per shift per face worker was 2902 to 3696 t. Coke production has now attained 1,800,000 t., as against 757,000 t. in 1919 and 3,523,000 t. in 1913, and the coke-ovens



consumed 2,343,000 t. of coal, of which 379,000 t. was imported. Owing to increased use on the Belgian railways, the output of briquettes rose from 2,458,000 t. in 1919 to 2,922,000 t. in 1920. Exports, largely to France, amounted to 1,638,000 t. of coal, 219,000 t. of coke, and 215,000 t. of briquettes; imports were 1,541,000 t. of coal, 124,000 t. of coke, and 152,000 t. of briquettes, Germany, Great Britain, and the United States supplying 1,211,000 t., 301,000 t., and 28,000 t., respectively. The total consumption of fuel amounted to 22,449,000 t., or 86 per cent. of the consumption in 1913.—(*Iron and Coal Tr. Rev.*, Mar. 25, 1921.)

**Position of the German Potash Industry.**—In reply to an inquiry made by the French delegates at the Brussels Conference, the German delegates stated that the German potash industry was now operating 201 shafts, 74 factories which produce chloride and sulphate of potassium, 8 special factories where salts from other works are treated, and also factories that work up by-products, such as bromine and magnesium chloride. Owing to the small demand and lack of coal, production was being still further reduced, especially in Hannover. The present production, however, could be more than doubled without sinking new shafts or erecting new plant, etc., and it could be increased many times were transport and labour conditions slightly better. Handling facilities were good, the works were all interconnected by rail, and adequate means were available for conveyance by water to the seaports.—(*Z. angew. Chem.*, Feb. 11, 1921.)

**The German Leather Industry in 1920.**—On the whole, 1920 was a good year for German leather manufacturers, but fine leather-ware was in little demand. When the industry was freed from Government restrictions in August, 1919, the importation of raw materials again became possible, and such war-time substitutes as wooden soles, paper fabric, etc., disappeared completely, although wooden heels are still made. As the export duty on leather products restricted the proper development of the export trade, the Government removed the duty so far as it affected the finer grades of leather and leather goods. The export trade fluctuated during the year and is still unsatisfactory, as the former German markets are well stocked and are making strenuous efforts to ward off German competition. The German leather factories use mainly lignite as fuel; they have suffered from frequent interruptions in the supply of electric light and power, and the increased railway rates, which became effective on December 1 last, will be acutely felt by the industry.—(*U.S. Com. Rep.*, Mar. 3, 1921.)

**Sugar Production in Europe.**—During the last six years the production of sugar in Europe has fallen by about 66 per cent., and Germany, formerly the largest exporter, cannot now supply her own needs. The European sugar production for the years 1913-14 and 1919-20 is given below:—

	1913-14.	1919-20.
	Million metric tons.	
Germany .. .. .	2-738	0-750
Austria-Hungary (former) ..	1-683	0-585
Holland .. .. .	0-231	0-233
Sweden .. .. .	0-137	0-145
Denmark .. .. .	0-146	0-160
Belgium .. .. .	0-229	0-127
France .. .. .	0-781	0-175
Russian Empire (former) ..	1-688	0-350
Other countries .. .. .	0-543	0-300
Totals .. .. .	8-181	2-830

Apart from political considerations, the large decrease in Germany is ascribed mainly to the high price of fertilisers, particularly nitrogenous fertilisers, which are chiefly used in great quantities for sugar-beet. Germany has lost important producing areas, but certain sugar factories in South Germany have formed an "Interessengemeinschaft" to further the development of the industry.—(*Schweiz. Chem.-Z.*, Mar. 1, 1921.)

**Indigo in Java.**—Indigo is grown on about 40 estates in Java as well as by the natives. The estate indigo contains from 60 to 80 per cent. of indigotin, but the native product, which is used locally, contains only 0.75 to 1 per cent. Before the war Holland took most of the powder and Singapore the paste, but since 1917 Japan has purchased the bulk of the powder. Singapore became an important market for the product during 1920.—(*U.S. Com. Rep.*, Feb. 19, 1921.)

**Drug Manufacture in Petrograd.**—During the period July–September, 1920, four State drug factories and four private factories were in operation. Two of the State factories produced:—Liquid medicines, 14,924 kg., powders, 7509 kg., ointment, 2444 kg., plasters, 65 kg., chemical and mineral preparations, 789 kg., chemical dressings, 1,500,000 kg., and iodine, 150 kg. The other two factories and the private laboratories manufactured sweetening material solely, the production in the above-mentioned period amounting to 196 kg. There was a general shortage of all raw materials, particularly fats.—(*Chem. Ind.*, Mar. 28, 1921.)

## PERSONALIA.

Dr. Morris W. Travers has been elected president of the Society of Glass Technology for the ensuing year.

The new chair of biochemistry at Cambridge University, to be known as the Sir William Dunn chair, has been offered to and accepted by Prof. F. Gowland Hopkins, fellow and prelector in biochemistry at Trinity College.

Mr. F. W. Harbord has been elected president of the Institution of Mining and Metallurgy in succession to Mr. Frank Merricks. Mr. Benedict Kitto, and Prof. R. Peele, of Columbia University, New York, have been elected honorary members.

Prof. E. Lagueur has been appointed professor of pharmacology and director of the Pharmacological Institute in the University of Amsterdam.

The eightieth birthday of Prof. K. Graebe, the discoverer of synthetic alizarin, was celebrated recently in his native town, Frankfurt-on-the-Main. Prof. Graebe occupied the chair of chemistry at Geneva University from 1878 to 1906, and last year he published a history of organic chemistry.

Prof. R. Scholl, of Dresden, has been appointed to the chair of chemistry at Vienna, in succession to Prof. Schlenk, who is to succeed Emil Fischer in the University of Berlin.

The following appointments, etc., at German Technical "Hochschulen" have been notified:—Prof. Schubert to be head of the chemical department at Charlottenburg, in succession to Prof. Dolezalek; Dr. L. Moser to the chair of analytical chemistry at Vienna; Dr. Alex. Classen is retiring from the chair of inorganic chemistry and electrochemistry at Aachen.

We record with regret the death, on April 10, of Mr. J. W. Leitch, the founder and head of John W. Leitch and Co., Ltd., Milnsbridge Chemical Works, Huddersfield.

The death is announced of Mr. John H. Yocum, president of Yocum Faust, Ltd., London, Ontario, a well-known leather chemist who joined this Society in 1894.

Prof. A. W. Reinold, formerly professor of physics in the Royal Naval College, Greenwich, died on April 11, at the age of 77 years.

The death is announced of Dr. P. Jannasch, formerly honorary ordinary professor of chemistry in the University of Heidelberg, aged 79.



## PARLIAMENTARY NEWS.

### HOUSE OF COMMONS.

#### *Farina Mills.*

In reply to questions put by Mr. J. Gardiner concerning a company formed to erect mills in this country for the production of farina, Sir P. Lloyd Greame said that the company was promoted by private individuals with Government support. The capital invested by the promoters was £20,000, and the cost of the four mills acquired at King's Lynn, Boston, Monikie, and Hull was £247,000. The King's Lynn and Boston mills had produced 1875 and 150 tons of farina respectively, but the Boston mill had been sold, and the other mills were on sale. Private subscribers were paid out at par.—(Apr. 11.)

#### *British Dyestuffs Corporation, Ltd.*

Sir P. Lloyd Greame, in reply to an inquiry by Mr. Hogge concerning the appointment of three new directors to the board of the above Corporation, stated that the appointment of directors (other than Government directors) to the board did not require the consent of the Government, as the latter had no controlling interest in the Corporation.—(Apr. 11.)

#### *Chemical Warfare Committee.*

Mr. Rawlinson asked the Chancellor of the Exchequer whether his attention had been called to the hardship caused to the members of the Chemical Warfare Committee by the Treasury rule that to obtain remuneration they must prove actual pecuniary loss due to their service on the Committee; and whether, as some of the members received fixed incomes or were engaged in research work, he would arrange that members entitled to remuneration should not be compelled to show actual loss.

Lieut.-Com. Young, in a written reply, said that members of commissions and committees were usually willing to serve their country without specific remuneration in cases where such service did not entail actual loss, and he saw no reason to depart from the usual rule in this particular case.—(Apr. 12.)

#### *Basic Slag.*

Sir A. Boscawen informed Sir J. Norton-Griffiths that the prices of basic slag, 84 shillings per ton from January to May, 1920, and now 127s. 6d. per ton, were maximum prices which the manufacturers, by agreement with the Ministry, undertook not to exceed. The higher price was sanctioned in view of the increased cost of transport and production, and because without such an agreement the price would probably have gone still higher. The agreement expired on May 31, 1921, and would not be renewed. Export of basic slag was prohibited, save under licence, as the supply of the higher grades was insufficient to meet home demands. The Government had never owned any basic slag other than the small quantities required for use in the Ministry's farm settlements or experiments. No licence was required for the importation of basic slag, and during the six months ending March 31, 1921, 15,769 tons, worth £125,832, had been imported, the bulk of which was probably unground.—(Apr. 13, 20.)

#### *Coal Industry (Capital, Profits, and Wages.)*

Mr. Bridgeman, answering Mr. Hurd, said that, exclusive of coke ovens and by-product plant, the total capital employed in the coal-mining industry was approximately £180,000,000, not including new money invested in the industry during 1920. The percentage of profits earned, before deducting profits carried to reserve and capitalised, was estimated

as follows:—Average of five years ended March 31, 1914, 9·8 per cent.; average 1915–1917, 17·0 per cent.; average 1918–1921, 15·5 per cent. During the seven years ended March 31, 1921, it was estimated that £40,000,000 was appropriated from profits to capital, leaving available for dividends, partners' drawings, and income-tax, a sum representing a possible yield of 11½ per cent. on the average capital employed. The total amounts of wages paid were £91,000,000 in 1913 and £261,721,000 in 1920; the profits in these two years were £21,000,000 and £27,000,000, respectively.—(Apr. 19, 22.)

## REPORTS.

GENERAL REPORT ON THE ECONOMIC SITUATION OF DENMARK AT THE CLOSE OF 1920. By R. M. TURNER, *Commercial Secretary to H.M. Legation, Copenhagen*. Pp. 54. (London: H.M. Stationery Office. 1921.) Price 1s. 3d. net.

The present unfortunate economic situation in Denmark is to a large extent due to the high prices that have to be paid for imported fuel, which is now obtained mainly from the United States. It is therefore not surprising that increasing interest is being taken in the development of home resources, in water-power schemes, electricity, and the extensive deposits of peat. A marked feature of the past year has been the depreciation of the Danish krone, which has been investigated by a Government commission but so far without result, although it may soon lead to a revision of the tariff system. Unemployment and the cost of living have both steadily increased, and the curtailment of credits by the banks has affected trade and industry.

The condition of agriculture, the main source of wealth, is favourable. Sugar-beet was cultivated over about 95,300 acres, or some 6400 acres less than in 1919, when the production was 135,000 metric tons; about 10,000 t. of the 1919 crop was exported. Experiments on the production of sugar-beet seed have so far not led to any tangible result, but the seed industry in general has been developed, and more potatoes have been cultivated and exported. The oil-crushing and margarine industries have been active, and it is noteworthy that the importation of edible oils has diminished, and that of oil-bearing nuts and seeds has increased. The fact that it is advantageous to export butter in order to improve the exchange augurs well for the prospects of these industries. Of margarine some 45,000 t. was produced during 1920, which is about 75 per cent. of the output capacity; and nearly all of it was consumed in the country, regulations as to packing, colouring, etc. effectually preventing exportation. Much of the raw material for the leather industry is obtained in the country; imports of heavy hides and tanning extracts and exports have decreased, but on the whole the industry, which is practically controlled by a single group, has been well employed. Shortage of fuel has seriously affected cement and brick manufacture, and the textile industry has been passing through a bad time owing to shortage of imported raw materials, fuel, and foreign competition. These causes, together with increased labour costs, have also affected the metal industry, which on the whole has been fairly well occupied, and the chemical industry, which, in addition, has had to contend with German competition. The manufacture of fertilisers has continued favourably notwithstanding importation of foreign superphosphate, etc., which depressed prices. During the first nine

months of 1920, 82,095 t. of raw phosphate was imported, compared with 35,698 t. for the whole of 1919, and the corresponding figures for superphosphate are 45,565 t. and 66,864 t., respectively. In the same period there was imported 42,234 t. of Chilean and Norwegian saltpetre, representing a decrease of 21,098 t. over the corresponding nine months of 1919, but the imports of the Norwegian fertiliser, 32,819 t., increased by 10,492 t. The importation of potash fertilisers decreased from 92,142 t. to 3126 t. for the nine-month period. Imports of glass and glassware, and of porcelain and faience (mainly from Germany) declined notably; and of metals there were imported increased quantities of cast iron (28,600 t.), black plates (44,000 t.), tinplate (6700 t.), and raw lead (3700 t.).

The main facts in the economic position to be noted by U.K. manufacturers are, apart from the question of exchange, the existence of large stocks in the country and foreign, especially German, competition.

**RAPPORT SUR LE COMMERCE ET L'INDUSTRIE DE LA SUISSE. 1919. Pages 495. Publié par le Vorort de l'Union suisse du Commerce et de l'Industrie. 1920.**

The year 1919 was a chequered one for the Swiss heavy chemical industry. Liquidation of war stocks and widely fluctuating prices combined to make markets unstable, and until stocks had been disposed of towards the middle of the year and the textile industry had resumed activity, production was not always able to meet demand. The shortened working day also restricted production.

The foregoing applied chiefly to sulphuric acid, but oleum was always in demand. Pyrites was in sufficient supply, but the price went up instead of down. Switzerland was fortunate in not having embarked on the manufacture of sulphuric acid from calcium sulphate, as this process has not fulfilled expectations. Lack of coal and sulphuric acid prevented over half the hydrochloric acid and sodium sulphate plants from working, in spite of the brisk demand; imported rock salt was used and hydrochloric acid was imported at prices below the production cost in Switzerland. The manufacture of nitric acid from atmospheric nitrogen by the arc process, which was actively pursued during the war, fell off, and as many consumers imported saltpetre or nitric acid from abroad, the factories diverted production to sodium and calcium nitrates. The soda industry, which suspended work at the end of 1918, recommenced on a small scale in June, 1919, and by the end of the year was able to meet requirements. In the fertiliser industry supplies of phosphates were no easier to obtain than during the war, and prices remained high; but little basic slag was imported, although potash salts were in plentiful supply. The electrochemical and electrometallurgical industries suffered under the prevailing economic depression, and many of the works had to strike out along new lines. Owing to the termination of war contracts only 2 or 3 of the calcium carbide factories that were running in 1918 continued to work. The following quantities of carbide were exported in the years 1913-1919 (in metric tons):—31,790, 35,950, 55,410, 58,010, 59,450, 75,840 and 36,890 t.

The manufacture of cyanamide was affected by a smaller demand and foreign competition, the output being reduced from 40,000 t. in 1918 to 9000-10,000 t. in 1919. Little improvement could be foreseen for the aluminium industry, which has suffered from the development of the industry in England, the United States, and Scandinavia. The production and export of ferro-alloys were very small. Some progress was made in the electric-smelting of iron

scrap, and the output was estimated at 16,000 tons; smelting direct from the ore would only be commercially feasible in Switzerland were electric current available at about 1 centime per kw., and this is not possible. The export trade in chlorates and perchlorates was extremely small, and the manufacture of perchlorates had to be discontinued. The market for caustic soda, chloride of lime, chlorine, and similar products was fairly good, and the manufacture of sodium, although not for export, was continued at the Monthey works of the Swiss Society of Chemical Industry, where it is used in the manufacture of synthetic indigo. The country's requirements in chlorine are covered by the output of the Monthey works, and the production of this gas has apparently become stationary. Manufacture of perborate was abandoned, as the current consumption was too great, but the output of other minor products, such as hydrogen peroxide and persulphate, improved. Electrolytic oxidation and reduction are used in the Swiss dyeworks, but details are not available.

From the middle of 1919 the demand for coal-tar dyes has been so great that the factories have not been able to meet it; the production could be increased many times were sufficient raw materials, especially coal, available. Exportation of dyewares to France in 1919 was very severely handicapped by the French official regulations and duties. No difficulty was experienced in exporting to England, where the Trade Licensing Sub-committee granted import licences freely. All the dye-works manufactured a wider range of colours, although not to the same extent as before the war. Production of synthetic indigo was restricted by the shortage of aniline oil; nevertheless, both production and sales were about twice as large as in 1918. Mainly owing to excessively high prices, to a great extent adventitious, the export trade in synthetic perfumes was very much better than in 1918, but, in spite of this, the outlook for the industry was held to be very doubtful. The mineral colour and varnish industry experienced a bad year as it was congested with stocks purchased at war-time prices and could not dispose of them. The manufacture of explosives was necessarily curtailed, and the future of this industry was not regarded cheerfully except in so far as the development of water-power resources would call for considerable supplies.

Of the many difficulties which confronted the manufacture of pharmaceutical chemicals, the reduction in the length of the working day was the greatest and had the most effect in raising production costs; in the latter part of the year firms which did not also manufacture dyes were scarcely able to make ends meet. Exportation was small and very difficult owing to collapsed exchanges; and the fairly satisfactory results which were obtained were due to the absence of foreign competition. According to one authority, the year 1919 was worse than any of the years of the war. The soap industry also recorded an unfavourable year. Large quantities of raw materials purchased at high prices during the war were delivered to the last kilogram, and at the same time buyers stood aloof waiting for prices to fall, and foreign wares, particularly Spanish, were thrust upon the market. The slump in the building trade naturally affected the cement industry which, in spite of lower prices, worked at only half-capacity. Manufacturers of porcelain and faience had to contend with importation from Germany and Lorraine, but makers of common pottery were in a better position, as their raw materials are found in the country. Glass manufacture was badly hit by the shortage of fuel; before the war the cost of fuel was 15-25 per cent. of that of the finished product, now it is about 60 per cent. The three glass-bottle factories produced regularly, and a small proportion of the output was exported.

Other subjects included in this comprehensive report are textiles, metals, engineering, preserved foodstuffs, leather, rubber, etc., together with much statistical information.

## GOVERNMENT ORDERS AND NOTICES.

**FORMER ENEMY ALIENS AND THE DYESTUFF INDUSTRY.**—The Board of Trade announces that by notice published in the *London Gazette* of April 8, the provisions of Section 11 of the Aliens Restriction (Amendment) Act, 1919, which imposes restrictions on the acquisition by former enemy aliens of certain kinds of property, have been applied to the following industries, namely, the manufacture of synthetic organic dyestuffs, colours, or colouring matters, and the manufacture of organic intermediate products used in the manufacture of synthetic organic dyestuffs, colours, or colouring matters. The effect of this is to make it unlawful for any former enemy alien to acquire either in his own name or in the name of a trustee any interest in the industries in question, or any share, or interest in a share, in a company registered in the United Kingdom which carries on the industries.

**ORDERS UNDER THE GERMAN REPARATIONS (RECOVERY) ACT, 1921.**—German Reparations Recovery No. 1 Order, 1921.—Any article imported before May 15 is exempted from the provisions of the Act if it be proved (a) that a contract was entered into before March 8, 1921, and (b) that not less than 20 per cent. of the purchase price was paid before the said date in pursuance of such contract, or (c) that the physical possession and property in the article, the subject of the contract, had passed to some person other than a German national prior to the said date.

German Reparations Recovery No. 2 Order, 1921.—This Order extends the provisions of Section 4 of the Act so as to include contracts for resale entered into by importers before March 8, 1921.

**PETROLEUM PRODUCTION (TRANSFER OF POWERS) ORDER, 1921.**—By an Order-in-Council the powers of the Ministry of Munitions under the Petroleum (Production) Act, 1918, and all rights and liabilities acquired or incurred by the Minister of Munitions under that Act, have been transferred to the Board of Trade, such powers to be exercised through the Secretary for Mines.

**EXPORT OF INDUSTRIAL EXPLOSIVES.**—The Board of Trade (Licensing Section) has issued an open general licence permitting the export, as from April 14, 1921, of the following explosives:—Rock-ite, super-cliffite No. 1, super-cliffite No. 2, super-rippite, tonite or cotton powder No. 1.

**CERTIFICATES OF ORIGIN.**—The following goods, *inter alia*, are exempt from the requirements of certificates of origin:—Marble, limestone, sandstone, ganister, sulphur, iron and copper pyrites, mechanical pulp, and iron and other metalliferous ores and scrap (except from Denmark, Holland, and Belgium). Declarations of non-German origin are still required.

## LEGAL INTELLIGENCE.

**BREACH OF CONTRACT FOR SALE OF SODA PRODUCTS.**  
*John Batt and Co., Ltd. v. Aniline Dye and Chemical Co.*

On April 12, in the King's Bench Division, Mr. Justice Shearman had before him in the form of

a special case the arbitration proceedings between John Batt and Co., Ltd., London, and the Aniline Dye and Chemical Co., which referred to the breach of a contract for soda ash and caustic soda.

Counsel for John Batt and Co. stated that his client's complaint was that the chemical company had broken its contract to sell to Batt and Co. 400 tons of soda ash and 100 tons of caustic soda; and that under the arbitration proceedings Batt and Co. asked for the difference between the contract price and the market price. The chemical company had failed to ship the goods. The point raised by the sellers was under the red ink clause in the contract, viz., "The price of goods sold under the contract may be advanced from time to time, and the present price of raw materials and the current rate of wages increased owing to the action of the Government; and the buyer shall have the option of saying whether on the advance he will accept it or refuse to have the goods." Counsel added that before the breach of the contract no notice was given by the sellers to the buyers.

His Lordship upheld the arbitrator's award, and ordered the chemical company to pay Batt and Co. £7050 and costs.

## COMPANY NEWS.

### UNITED ALKALI CO., LTD.

Addressing the adjourned twenty-ninth and the thirtieth ordinary general meetings, held at Liverpool on April 20, Mr. Max Muspratt, the chairman, said that difficulties had been enormously accentuated by the greatly increased cost of all construction, the slowness of delivery of plant, and the high cost of production, especially of fuel and labour. The first had led to an expenditure of several hundred thousand pounds in excess of estimates; the second had delayed the complete operation of more economical processes in time to meet the boom demand; and the third had reduced profits to a minimum. Since trade had fallen off production had been curtailed, and at the present time several of the company's works were completely, and others partially, shut down. On the other hand, deferred repairs had been overtaken and new processes introduced. The Leblanc soda process had been superseded by the electrolytic process. The discovery of brine at Widnes indicated an improvement in the company's position, and since the beginning of this year brine from this source had supplied all the electrolytic plants.

Dealing with finance, Mr. Muspratt stated that during the last six years the company had paid £1,759,233 in taxation, as against £1,847,905 in net dividends, and that £2,500,000 had been spent on plant in accordance with instructions from the Ministry of Munitions. By making certain adjustments of the debenture capital it was hoped to avoid offering any new share capital for public subscription. In spite of considerable profits made in 1920, it was considered unwise to pay a dividend on the ordinary shares. In view of the falling costs of living, proposals had been made to the employees that they should accept reductions in wages. The company was now producing on the best and most economical lines, and a comparatively small increase in the demand for its products would put it on a sound basis of profitable manufacture.

Dr. G. C. Clayton stated that, owing to the coal strike, the works were rapidly starving for coal, and in the course of a few days would have to close down.

### THE BRITISH PORTLAND CEMENT MANUFACTURERS, LTD.

At the tenth ordinary general meeting, held in London on April 12, the Hon. F. C. Stanley, chairman, said that in 1920 production, deliveries, and profits of cement had all been better than in any year since the outbreak of war. The difficulties attending manufacture and distribution became less severe during the latter part of the year, and though at the moment there was more than a lull in business, there were indications that the tide had turned, and he looked forward to a revival in the demand for cement. The demand had been very acute during the whole of the past year, and every effort had been made to meet it; if manufacturing cost was high, the average price was good and the profits satisfactory, especially in the export markets. Although prices in the export markets were far above those ruling at home, the company had voluntarily restricted its export sales in order that the great home demand might be satisfied. The output had gradually improved during the year, and at its close was approximately equal to that of the last pre-war year. The Cement Marketing Co., which was formed in 1920 to distribute the company's production and that of certain allied companies, had fully justified expectations.

Whereas the balance of profit brought forward from 1918 was £136,264, subject to excess profits duty, that now carried forward was £181,477, after deducting an ample sum for the duty and the corporation profits tax. The trading profit, less repairs and renewals, taxes, etc., was £558,618, and after meeting debenture interest, etc. and putting £150,000 to general reserve, the balance was £323,406 (issued capital, including debentures, £3,698,080). Owing to the need for conserving resources, the dividend for the year on the ordinary shares would be limited to 15 per cent. (8 per cent. for 8 months in 1919). The capital account had been increased by a large sum representing the value of the three subsidiary undertakings (Tolhursts Cement Works, Ltd.; Trechmann, Weekes and Co., Ltd.; and the Artillery and Albion Cement Co., Ltd.) which had been transferred from the investment account, and a heavy mortgage on one of these had been paid off. In addition, the expenditure of £237,000 on works and properties had been added to the fixed capital account, and provision would have to be made for the works now being erected in India. Owing to these considerations it had been deemed advisable to issue to a financial group the whole of the remaining debenture stock, viz., £509,533, on the basis of an annual yield of 7½ per cent.

### SHAWINIGAN WATER AND POWER CO.

The report of the Shawinigan Water and Power Co. states that the capacity of the company's power stations is now about 220,000 electrical h.p., compared with 20,000 h.p. in 1905. The company began operations in 1900 and during the last five years the load has grown at an average rate of 20,000 h.p. per annum. A further 40,000 hydraulic h.p. will be developed in 1921 and this will lead to an increase in the company's revenue of over \$1,000,000 per annum. The output of 1,140,759,076 kw.-hr. in 1920 is only paralleled by one or two other industrial centres in North America. The assets of the company are valued at \$34,000,000, mortgage bonds etc. at \$13,000,000, and reserves amount to \$4,000,000, or, roughly, 20 per cent. of the issued share capital.—(*Canad. Chem. and Met.*, Mar., 1921.)

### OFFICIAL TRADE INTELLIGENCE.

(From the Board of Trade Journal for April 14 and 21.)

#### OPENINGS FOR BRITISH TRADE.

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W. 1, from firms, agents, or individuals who desire to represent U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number:—

Locality of firm or agent.	Materials.	Reference number.
Australia .. ..	Pottery .. ..	*412 26/
Canada .. ..	Drugs, etc. .. ..	7531
" .. ..	Olive oil .. ..	458
" .. ..	Pharmaceutical chemicals .. ..	401, 483
" .. ..	Glass, china .. ..	468
" .. ..	Drugs, etc. .. ..	469
Belgium .. ..	Ink .. ..	497
" .. ..	Machine and industrial oils .. ..	498
Denmark .. ..	Sugar .. ..	503
Hungary .. ..	Skins, tanning materials, leather .. ..	507
Italy .. ..	Agricultural chemicals, fertilisers, soap, varnish, dyes .. ..	473
" .. ..	Tinplate, black plates, terne plates .. ..	474
Netherlands .. ..	Fine chemicals .. ..	509
Poland .. ..	Tinplate .. ..	510
Spain .. ..	Chemicals, pharmaceutical products, shoe leather .. ..	476
Switzerland .. ..	Rubber tyres .. ..	477
Mexico .. ..	Photographic materials .. ..	516

\*The Official Secretary, Commercial Information Bureau, Australia House, Strand, London, W.C. 2.

†The High Commissioner for Canada, 19, Victoria Street, London, S.W. 1.

**MARKETS SOUGHT.**—A Canadian tannery has for disposal 5000 sides of harness leather from 14 to 30 lb. to each side.

A Canadian tannery offers 30 tons of medium wax splits, all shorts, 50 per cent. seconds. [Inquiries to the Canadian Government Trade Commissioner, 73, Basinghall Street, London, E.C. 2.]

#### TARIFF. CUSTOMS. EXCISE.

**Australia.**—The application of the new customs duties on soda ash and caustic soda has been postponed until October 1, 1921, and those on iron and steel tubes and pipes [Tariff Item 152A] until January 1, 1922.

**Belgium.**—The customs duties have been modified on, *inter alia*, gold, silver, platinum, perfumery, pottery, porcelain, glassware (except bottles, vials, demijohns and carboys).

The maximum "coefficient of increase" which the Government is authorised to apply has been raised to 6; articles upon which it has been increased include candles, cocoa, yeast, matches, certain metals, paper, cardboard, certain hides and skins, liquid carbonic acid, acetic and sulphuric ethers, soap, glass bottles, vials, demijohns, and carboys.

**British India.**—The complete text of the revised schedule of customs duties is set out in the issue for April 14.

Among the articles that are free of import duty are salt for industrial use, raw hides and skins, metallic ores, certain oilseeds, fertilisers, woodpulp, paper-making materials, quinine, and trade catalogues (by post).

Articles that pay duty at special rates include sugar, salt, petroleum, opium, and matches. Other goods pay *ad valorem* duties varying from 2½ to 20 per cent. The duty is 11 per cent. on chemicals,

drugs, medicines not otherwise specified, dyeing and tanning materials, paints, colours, glass, earthenware, china, leather, paper, cardboard, oils, oil-cake, perfumery, pitch, tar, soap, starch, and candles.

**Canada.**—Recent customs decisions affect circular sheets of steel, and aluminium, copper, brass, zinc, and other metal circles.

**Chile.**—The new tariff modifications came into force on February 23. The duty is unchanged on condensed milk, raw cocoa, sugar, petrol, paraffin wax, mineral oils (not specially mentioned), prussian blue, and ultramarine.

Among the articles on which the duty has been doubled are chocolate, vinegar, perfumery, and perfumed soap.

As from February 14 the import of opium, coca, cocaine, and substitutes thereof is subject to licence from the Director-General of Health.

**Egypt.**—Groundnuts and helba may now be exported without special licence.

The tariff valuations of iron and steel manufactures with effect from March 16 to May 15 are given in the issue for April 21.

**France and Algeria.**—The import duties have been modified on aluminium, celluloid, and bottles.

**France.**—The prohibition of the export of soap has been withdrawn.

**Germany.**—The import of oleomargarine and of premier jus is no longer subject to licence.

**Do. (Occupied Territory).**—The full text of the customs organisation and import and export regulations is set out in the issue for April 21.

**Gold Coast.**—An export duty of 1d. per lb. has been levied on kola nuts exported by sea, with effect from February 4.

**Greece.**—Caustic soda may again be imported. The export of olive oil of the present crop is permitted subject to a special licence.

**Mexico.**—Crude mineral oil, sulphuric acid, blasting powder, and other explosives not specially mentioned may be imported duty free as from March 14.

**Montserrat.**—Calcium arsenate may be imported duty free.

**Netherlands.**—Bones, fertilisers, and sugar may now be exported.

**New Zealand.**—As from January 27 the import of opium, morphine, heroin, and cocaine is subject to permit from the Minister of Customs.

**Norway.**—The export of all goods is permitted except, *inter alia*, explosives, bones, cellulose, wood pulp, copper ore, cyanamide, gold, silver, hides, skins, loather, nitrate of lime, paper, cardboard, and superphosphates.

**Palestine.**—The import of opium and saltpetre is subject to special licence.

**Portuguese East Africa.**—A copy of the new customs tariff for Manica and Sofala may be seen at the Department, 18, Queen Anne's Gate, S.W. 1.

**Spain.**—The import duty on sugar has been raised to 60 pesetas per 100 kg. as from March 30.

As from April 1 the import duties on the various classes of paper which were suspended in August last have been reimposed.

**Sweden.**—Export prohibitions have been withdrawn from certain hides and skins, etc.

**Switzerland.**—The import is prohibited, except under licence as from April 8, of thick leather, calf leather, and other leather for boot and shoe uppers.

**United States.**—Articles affected by the proposed emergency tariff legislation include olive oil, groundnut oil, sugar, molasses, syrup, flax-seed, and condensed milk.

## TRADE NOTES.

### FOREIGN.

**St. Vincent in 1919.**—Cotton was planted on 6633 acres, the largest area ever recorded, and the production was 212 tons, compared with 195 t. in the previous year. The sugar-cane industry is resuming its former importance and the manufacture of syrup has increased considerably; but very little sugar is produced. The production of syrup and molasses was 170,842 gallons. Owing to low prices and large stocks, the area under arrowroot has decreased; only 1002 tons was exported, compared with 2225 t. in 1918. There was also a decline in the area under groundnuts, of which 11,329 bushels was exported. The Agricultural Department has continued its work on cotton breeding, on the production of supplies of pedigree Sea Island cotton seed, and on the manuring and planting of cotton and arrowroot. The exports, valued at £149,749, included:—Arrowroot, £42,222; cotton, £66,782; syrup and molasses, £10,726; cassava starch, £4836; and groundnuts, £5098. Over 53 per cent. of the exports was taken by the United Kingdom, 27 per cent. by the British West Indies, and 8.7 per cent. by Canada. Of the imports, valued at £185,128, the United Kingdom, United States, and Canada supplied 31.4, 29.2, and 29.6 per cent., respectively (*cf. J.*, 1920, 404n).—(*Col. Rep.—Ann.*, No. 1063, Feb., 1921.)

**German Exports to England.**—German exports to England increased greatly during the last six months of 1920; during the third and fourth quarters of the year they included:—Dyestuffs, £1,000,000, £3,040,000; glass bottles, £127,000, £164,000; crude zinc, £67,000, £315,000; and raw sugar, £12,000, £240,000.—(*Handelsmuseum*, Mar. 31, 1921.)

**The Japanese Dye Market.**—Increased activity followed the introduction of the new import tariffs in August last, but so long as the weaving sheds are working short time it is unlikely that consumption will equal supply. Towards the middle of November there was a general rise in prices with the exception of Malachite Green, Sulphur Black, and Basile Blue. The suggested amalgamation of Japanese dye makers will probably not materialise; some of them, apparently, only desired it in order to avoid liquidation.—(*Chem. Ind.*, Mar. 28, 1921.)

**Foreign Company News.**—**Poland.**—The Polish Dyeworks Company, recently founded in Wierzbincin-Krainski, near Bromberg, has purchased a disused factory, and will manufacture mineral colours, lakes, and varnishes.—(*Chem. Ind.*, Mar. 28, 1921.)

**Holland.**—The Netherlands Dyestuff Factory has been formed in Amsterdam by certain Dutch banks and chemical firms to manufacture and trade in dyewares and other chemical products. The capital is 5 million florins (florin=1s. 7.8d. at par).

**Germany.**—The new Distillers' Yeast Syndicate, founded in March, represents nearly 95 per cent. of the German production, the combined output of its constituent firms being about 37,500 metric tons. The syndicate (Brennerei-Hefesyndikat G.m.b.h.) will fix prices and control sales, and it is expected to be much stronger than the former combine.—(*Handelsmuseum*, Mar. 24, 1921.)

**France.**—The Cie. Industrielle des Verreries à Vitre (capital 14.9 million fr.) is building large works at Cusset, in Allier, to manufacture window-glass by the Fourcault process on a very large scale. The company has taken over the Verreries Valentin Mesiner at St.-Etienne, and has also acquired the Aniche works of the Société Industrielle de Verrerie.—(*Bd. of Trade J.*, Mar. 24, 1921.)

**Japan.**—The formation of a combine of Japanese producers of sulphur colours, initiated by the Teikoku and Kompira dye companies, has made further progress, and a commission has been appointed to investigate the financial status of 18 firms producing sulphur colours in Tokyo, of which the total paid-up capital is about 4 million yen. If the combine is effected a new undertaking will be formed with a capital of 6 million yen.—(*Chem. Ind., Mar. 21, 1921.*)

**United States.**—A new company entitled "The Industrial Potash Corporation" has been formed with a capital of 30 million dollars to exploit the extensive alunite deposits near Marysvale, Utah, and to erect a plant to treat some 10,000 t. of material daily, mainly with a view to producing potassium sulphate for agricultural purposes.

It is reported that the Du Pont Co. is considering plans for the erection of a dry-colour plant to be the largest in the world, and to add a new paint and varnish plant to the existing units.

The capital of the Allied Chemical and Dye Corporation is to be increased from \$48,043,675 to \$113,043,675.—(*Oil, Paint and Drug Rep., Mar. 14, 1921.*)

**Norway.**—With the exception of the cellulose factories and the Norsk Hydro-Elektrisk Kvaestof A.-S., Kristiania, which manufactures Norwegian saltpetre and ammonium nitrate, all other chemical and allied undertakings have been forced to curtail production or to close down. Three firms in the carbide industry have ceased to produce and the remainder are working at 30–40 per cent. capacity. About 40 per cent. of the mines is being worked. The Glomfjord zinc-smelting works, a private undertaking run largely with Swedish capital, has suspended operations, and it is reported that negotiations for its sale to English interests are proceeding.—(*Chem. Ind., Mar. 14, Apr. 11, 1921.*)

**Sweden.**—The net profit for 1920 of the Svenska Tändstiftsfabriks A.-B., which comprises the majority of the Swedish match factories, was 12,950,000 kronor (krona=1s. 1½d. at par), and the dividend 14 per cent. (capital 45 million kr., reserves 65 million kr.). The production of 600,000 cases represents about 20 per cent. of the world's output; the turnover was 105 million kr., of which 95 million kr. was in respect of foreign sales. Prospects are now more favourable, as Japanese competition is much less keen than it was before the war. Owing to the high exchange value of the yen the Trust has been able to capture the Indian market, and in Australia racial feeling has told against the Japanese. The Trust anticipates that by April its stocks will have fallen to a normal level.

The liquidation is announced of the well-known oil-mill and soap works Malmö Oljeslageri and Hap-pachs Sappfabriks A.-B., Malmö, and at the same time the formation of a new company bearing the same title with a capital of 1.5 million kr. The Barnängens Kemiska Fabriker in Stockholm and Hudiksvall has also failed, but work is to be continued. The projected absorption by Lever Bros., Ltd. of various Swedish soap factories may be regarded as definitely abandoned, but the formation of a combine within the country which will command about 90 per cent. of the entire production will be shortly effected. Companies producing superphosphate, paper, sulphite pulp, and iron ore have announced a partial or total suspension of operations. Unemployment is greater than during the early part of last year.—(*Chem. Ind., Mar. 18, Apr. 11, 1921.*)

**The Soap Industry in Argentina.**—In 1910 there were 200 soap factories in Argentina, having a total capital of £210,214, with an output valued at £602,860, and utilising imported raw materials worth £114,789. Since then the demand for soap

has increased, production has risen, and importations have declined from 1795 metric tons in 1913 to 1371 t. in 1919. Hard yellow soaps are mainly produced as they are in greatest demand, but their quality is poor. With the exception of animal fats, all the raw materials are imported. In 1919 the United States supplied nearly all the rosin, nearly one-fifth of the soda ash, over 70 per cent. of the caustic soda, and about 5 per cent. of the sodium silicate. American silicate of soda has been criticised on grounds of quality and price, and the European product is preferred. The total importations in 1919 were:—Rosin, 15,503 metric tons; soda ash, 3459 t.; sodium carbonate, 1570 t.; Solvay soda, 5878 t.; caustic soda, 6543 t.; and sodium silicate, 4512 t. There is a steady demand for coconut and palm oils, perfumes, and colouring matters.—(*U.S. Com. Rep., Feb. 18, 1921.*)

**The Phosphate Industry in Curacao.**—According to *De Telegraaf* of March 8, the position of phosphate mining in Curacao is favourable, despite the emigration of a number of miners to Cuba. During 1920 64,969 tons was exported, distributed as follows:—Sweden, 28,505 metric t.; Germany, 15,758 t.; Holland, 10,566 t.; Spain, 2946 t.; England, 2113 t.; and Norway, 508 t. Exports during previous years were:—1915, 31,308 t.; 1916, 14,468 t.; 1917, 3582 t.; 1918, nil; and 1919, 19052 t.—(*Official.*)

## REVIEWS.

**APPLICATION OF DYESTUFFS TO TEXTILES, PAPER, LEATHER AND OTHER MATERIALS.** By J. MERRITT MATTHEWS. Pp. vi.+768. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1920.) Price 10 dollars.

English readers have had abundant opportunity during recent years of becoming familiar with American publications on technical subjects, and will find that the present volume displays most of the points of excellence which they have learned to expect. The book is admirably bound, printed, and illustrated, but the text does not approach the standard of those English works on the subject with which it challenges comparison. The major portion of the work deals with textiles and includes sections on the chemistry of the textile fibres and the processes of scouring, bleaching and the like, as well as of dyeing. The treatment of these subjects is such as to form a suitable groundwork of instruction for pupils in technical schools. Each chapter concludes with an account of experiments which are very well devised for illustrating and impressing on the minds of students the points dealt with in the earlier part of the chapter, and the whole collection of experiments would form an admirable laboratory guide in the study of the subject. The arrangement of subjects in the text is such as to involve frequent repetition of the same statements, although with a different lay-out of the subject matter considerable space could have been saved. The details of works practice and the special precautions to be taken in work on a large scale are very inadequately treated. This is the more to be regretted because the book contains more numerous and excellent illustrations of machinery than the writer remembers having seen in a work on this subject. These illustrations, however, are for the most part not referred to in the text, and many of them are quite outside the scope of the work. Regarded as a text-book for students, the book appears to be rather too dogmatic and too little suggestive and stimulating to



thought. The author is disposed to give explanations which strike one as merely facile. Such explanations may have some value as mnemonics, but it is doubtful if they are calculated to encourage a scientific habit of mind or lead to a sound knowledge of the subject. The treatment of such subjects as the application of sulphur colours and of mordant dyes on silk leaves a general impression that the dyeing industry in America has not reached so advanced a stage as in this country.

The book contains some useful summaries of the special properties of dyestuffs, and much information which can be found elsewhere only in a scattered form is here collected together very conveniently for reference. There is a copious bibliography.

In conclusion, this book can be recommended as a useful addition to technical libraries on account of its excellent illustrations and summaries as well as for the admirably devised series of illustrative experiments.

BENJAMIN LERCH.

#### WATER PURIFICATION PLANTS AND THEIR OPERATION.

By MILTON F. STEIN. *Second edition*. Pp. 270. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1919.) Price 16s. 6d. net.

The second edition of this book has recently appeared, and contains new chapters on bacteriological tests for water and their interpretation.

The fact that the book deals almost exclusively with American waterworks, where the problems to be faced are to a great extent different from our own, very considerably curtails its usefulness in this country, and the further fact that the chapters on chemical and bacteriological tests and their interpretation are written by an engineer for non-technical men is also a decided disadvantage. It certainly does not seem wise that the chemical and bacteriological tests of such an important commodity as drinking water should be entrusted to anyone but a trained chemist or bacteriologist, and only a sanitarian should have the duty of interpreting them. The risk involved is too great; an error in carrying out the tests or a faulty interpretation of the results might open the way to very considerable trouble. Apart from this general principle, the author's treatment of this part of the subject leaves much to be desired. No mention is made in the chemical tests of any test for organic impurity such as free and albuminoid ammonia, or oxygen absorbed from permanganate, which are still insisted on by English sanitarians, and which are undoubtedly of great value when properly interpreted. The bacteriological tests given are also somewhat crude, although, as in the chapter on chemical tests, elaborate details are given of the apparatus required. The tests consist of the gelatin and agar counts and the litmus-lactose-agar, and lactose-broth tests for intestinal microbes. The latter tests would include many non-intestinal microbes, and to this extent err on the side of safety, but to the scientific mind something more exact would be preferable. A word may here be said on the difference between English and American standards. The Americans seem to be content with a standard based on percentage reduction, whilst in this country an absolute standard is generally insisted upon. Thus, if the raw water contained, say, *B. coli* in 0.001 c.c., and in the finally purified effluent this was reduced to a positive result with 1 c.c., they would call this a 99.9% reduction and be satisfied with it, but in this country we are not completely satisfied with sending water into consumption even with *B. coli* in 100 c.c.

This review has dealt chiefly with the chemical and bacteriological parts of the book, as they would seem to be those of most interest to members of the Society of Chemical Industry, and to that extent is adverse criticism: but much of the book is excellent.

Following an introductory chapter on sources of supply, a long chapter is devoted to detailed engineering descriptions of typical plants in the United States, covering all methods of purification. After the chapters on chemical and bacteriological tests, coagulation and sterilisation are carefully dealt with. The theory of coagulation and the chemical reactions of the coagulants are discussed, and numerous plants for the addition of chemicals are described. Then follow interesting chapters on water-softening, sedimentation, filtration, and general operation, and some graphic charts for calculating quantities of coagulants, etc. required under diverse conditions. These, by the way, are drawn for U.S. gallons, which are smaller than the imperial gallon.

The book concludes with appendices dealing with the analysis of coagulants, instructions for making standard solutions, and specifications for certain chemicals. It is profusely illustrated with sketches, photographs and working drawings, and is written in a clear and readable style.

D. B. BYLES.

RUBBER, RESINS, PAINTS AND VARNISHES. By R. S. MORRELL and A. DE WAELE. *Industrial Chemistry Series*, edited by S. RIDEAL. Pp. xii+236, with 33 figures in the text. (London: Baillière, Tindall and Cox. 1921.) Price 12s. 6d. net.

This is yet another of the voluminous stream of recently published books belonging to what one is tempted to classify as the "Science-in-a-Hurry" type; books which, unfortunately, bear the obvious marks of hasty compilation and of lack of the care which is to be expected in scientific text-books.

The first part of this book deals with rubber, and occupies only 34 pages. Even allowing for unavoidable defects due to the extreme brevity of this section, one is compelled to state that it is lacking in accuracy and that the information given is not so well selected or arranged as it might have been. Considering the great importance of rubber both from the scientific and technical points of view, it is perfectly obvious that nothing adequate could have been written in so small a space, and that this subject should have been given a volume to itself. The remaining sections of the book dealing with drying-oils, resins and pitches, pigments and paints (including linoleum), and varnishes, are more satisfactory, though at times lacking in discrimination of the relative importance of the different subjects dealt with. Thus, it is difficult to understand why, in an elementary industrial handbook, more than six pages are devoted to the analysis of varnish (a complex subject which is obviously dangerous ground to the student), whereas less than three pages are given to the analysis of oils, and paint-analysis does not appear to be even mentioned.

This volume, in common with others of the series, is apparently intended largely for the advanced student, and it will certainly be of some value to readers requiring to obtain with a minimum expenditure of time and labour a general idea of the subjects dealt with. It is, perhaps, open to discussion whether such books are not somewhat dangerous to the student, as being calculated to lead him to accept short-cuts and easy paths to knowledge when he ought to be learning how to consult standard books and original papers. One must, however, admit that the authors have made

some allowance for this in giving a short but useful bibliography of the subjects and fairly frequent references to journals, etc.

The reading of this book (particularly the first part) brings to notice a too-frequent fault in chemical and technical literature, i.e., the misquotation and mis-spelling of botanical names; there is no more excuse for these faults than there is for errors in chemical formulæ. Authors and publishers would do well to attempt to obtain accuracy in botanical nomenclature.

RUSSELL G. PELLY.

JOURNAL OF INDIAN INDUSTRIES AND LABOUR. Vol. I., Part I. February, 1921. (Calcutta: Superintendent, Government Printing, India. 1921.) Price Rs. 1-8 per part, or Rs.4-8 per volume of 4 parts.

The publication by the Government of India of a new journal devoted to Indian industries and labour is further evidence of the increased attention which industrial development is now receiving in that country. In a "Foreword," Sir Thomas Holland explains that the Journal has been established to function as a bond of union between those responsible for the machinery of development in the different provinces of India in which policy and natural resources may also be different. At the same time it is also intended to act as "a medium for communicating to a wider public, within and outside India, information that will assist private enterprise."

To the number under review Sir C. E. Low contributes a particularly interesting article on "Possibilities of Industrial Development in the Central Provinces and Berar." Although chiefly concerned with the future outlook for these territories, many of the difficulties and problems dealt with may be taken as affecting the industrial development of India in general. Mr. B. M. Das, writing on "Researches in Tanning and the Calcutta Research Tannery," describes the organisation and objects of an institution which should do much to place this industry, of so great importance to India, on a sound scientific basis. Dr. J. Coggin Brown's "Trade Notes on Bauxite," whilst applicable more particularly to Indian conditions, are also of general value. Mr. S. T. Gadre describes the results of an investigation on "Clove Oil from Clove Stems," and discusses the possibility of carrying out the process of extraction on a commercial scale in India. An article by Capt. G. N. Frankau on "The Gilt Wire and Tinsel Industry at Burhanpur" affords an illustration of an old industry, by no means the only example in India, which has fallen into decay chiefly through the antipathy of the worker to adopt machinery and modern methods and thus meet European competition. Papers dealing with "Welfare Work in Bombay Cotton Mills," "Trade Disputes in Bengal," and "Industrial Education in Madras Presidency" are all of interest as serving to show that the development of industry in the East has given rise to problems similar to those which are still occupying the attention of the Western world. The Journal also contains a useful summary of Industrial Intelligence from the various districts and some Miscellaneous Notes.

In addition to the Journal, the Government of India announces the publication of Bulletins of Indian Industries and Labour, uniform in size with the Journal, and intended to deal with specialised subjects in monograph form.

It may be confidently predicted that if the high standard set by the first number of this Journal is maintained, the objects of its publication as set out therein will be fully achieved.

A. RULE.

## OBITUARY.

### A. WYNTER BLYTH.

Alexander Wynter Blyth was educated at King's College, London, and practised as a physician for a few years in Worcester before he became Medical Officer of Health and Public Analyst for the County of Devon. From Devonshire he came to London and filled a similar position to the Borough of St. Marylebone. Associated for many years with the Sanitary Institute, he served on its Council, was chairman of its judges, and latterly hon. registrar. A past-president of the Society of Medical Officers of Health, he was for some years editor of "Public Health," and had much to do with the foundation of that journal. He contributed papers on sanitary science, law (he was also a barrister-at-law), and the chemistry of foods and drugs to the learned societies, and was the author of two well-known books, "Foods: their Composition and Analysis," and "Poisons, their Effect and Detection."

Wynter Blyth died on April 1 last in his seventy-seventh year.

## PUBLICATIONS RECEIVED.

TECHNICAL RECORDS OF EXPLOSIVES SUPPLY. 1915-1918. No. 1, DENITRATION OF SPENT ACIDS. Ministry of Munitions and Department of Scientific and Industrial Research. (London: H.M. Stationery Office. 1920.) Price 12s. 6d.

RELATIVITY AND THE ELECTRON THEORY. By E. CUNNINGHAM. Second edition. Monographs on Physics, edited by Sir J. J. THOMSON and Prof. F. HORTON. Pp. 148. (London: Longmans, Green and Co. 1921.) Price 10s. 6d.

THERMODYNAMICS AND CHEMISTRY. By F. H. McDUGALL. Pp. 391. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1921.) Price 30s.

THE FORMATION OF COLLOIDS. By THE SVEDBERG. Monographs on the Physics and Chemistry of Colloids. Pp. 127. (London: J. and A. Churchill. 1921.) Price 7s. 6d.

CRITICAL MICROSCOPY. By A. C. COLES. Pp. 100. (London: J. and A. Churchill. 1921.) Price 7s. 6d.

SAND-LIME AND OTHER CONCRETE BRICKS. By H. O. WELLER. Building Research Board, Special Report No. 1. Department of Scientific and Industrial Research. (London: H.M. Stationery Office. 1921.) Price 3d.

THE WINNING, PREPARATION, AND USE OF PEAT IN IRELAND. REPORTS AND OTHER DOCUMENTS. Fuel Research Board, Department of Scientific and Industrial Research. (London: H.M. Stationery Office. 1921.) Price 3s. net.

PUBLICATIONS OF THE UNITED STATES GEOLOGICAL SURVEY. Department of the Interior. (Washington: Government Printing Office. 1921.):—TUNGSTEN IN 1918. By F. L. HESS.

SALT, BROMINE, AND CALCIUM CHLORIDE IN 1919. By H. INSLEY.

MICA IN 1919. By H. INSLEY.

COAL IN 1918. Part B. Distribution and Consumption. By C. E. LESHNER.

ANALYTISCHE CHEMIE. By DR. TH. DORING. Wissenschaftliche Forschungsberichte, Naturwissenschaftliche Reihe, edited by R. E. LIESERGAU. Pp. 97. (Dresden and Leipzig: Theodor Steinkopff. 1921.) Price 3s. 5d.

## POST-GRADUATE TRAINING IN INDUSTRIAL CHEMISTRY.\*

FRANCIS H. CARR.

In recent discussions concerning the training of chemical students for industry there is to be traced a unanimity of opinion as to the need of modifying or extending existing courses of instruction, at least so far as concerns the education of certain classes of chemists required in the industries, especially the fine chemical industries. Notwithstanding this, it seems that too little is being done to bring about the necessary changes. The difficulty of finding the money required for equipment and endowment is illustrated in the case of the Department of Chemical Technology of the Imperial College of Science and Technology. In my humble opinion this college needs an expenditure of several hundred thousand pounds in order that the Department may properly fulfil its great national purpose. The difficulty of getting money does not lessen the need of it, but imposes upon those in whom the war has awakened the full realisation of our shortcomings the serious duty of explaining our needs.

A strong alliance between the universities and the industries is now regarded by all as advantageous to both. The industries have in the past undervalued the importance of the part which the universities play in the fulfilment of the national purpose, by aiding the development and progress of industry. The universities depend largely upon the demand for training created by industry, and a little thought must convince all that even the pursuit of pure research must ultimately depend upon the value of its results to the physical, mental or moral well-being of humanity. On the other hand, the future development of industry lies with the trained and selected men from the university, and in the special case of the chemical industry, much depends upon the work done by the university in giving the right direction to science students, and providing the right training for each type.

If a discussion becomes discursive, it is apt to prove barren; I will make, therefore, a few hypothetical assumptions and rule these out of the discussion. The first of these assumptions relates to the curriculum for graduation in science; although I would like to see this modified in minor details, let us assume for the moment that it constitutes the desired foundation for those taking up a career in chemical technology. Secondly, let us assume that a three years' course with chemistry as the principal subject does not qualify a man in technical chemistry. It is regrettable that there should be a general feeling abroad, especially among parents, that graduation, which normally occurs in the third year, implies fitness for practice. A student has to work hard enough in all conscience during his three years in order to merit a first-class B.Sc., without spending any time upon the cognate subjects required to qualify him as a technical chemist. It becomes, therefore, a question of post-graduate training. Do we need the colleges to undertake this?

There are those who hold that since some firms are willing and able, and indeed prefer, to train their own men, there is consequently no need for intra-mural training in chemical technology. My comment upon this is that there are only a few firms fitted to undertake this work, and these few certainly are not willing to train men for the many other firms who cannot undertake to do so; and

it is these unfitted firms which offer the greatest scope to the young technical chemist. The number of young chemists who are fortunate enough to gain admission to the works in which good training may be found is insufficient for a rapidly-developing industry, and even these men would gain much by attending a properly constituted course prior to the works training referred to. It is not all, or even most, of the chemists employed in works who need a special technological training, and only a small proportion of those who adopt chemistry as a career have the real instincts of the chemical technologist. Chemists not possessing this instinct may excel as workers in one or other of the works' laboratories, whether it be in that concerned with process-control, research or analysis. The technical chemist needs other qualities, such as ability to give directions when a reaction takes a course different from that expected, ability to accumulate experience of practical detail, to make yields higher and higher, to reduce the cost of labour more and more, and carefully to harvest from daily observation all those deductions which become summarised in a perfected process. Such power of controlling complicated and delicate processes, not merely conscientiously, but with intelligence and initiative, of observing closely and appreciating what in a mass of observed details is of practical importance, demands qualities of mind which are different from, though not incompatible with, those of the research chemist, although both types usually excel in the laboratory. These qualities are inbred, you will tell me, and are not produced by education. True enough; but the development of these qualities may be enormously helped by a training in certain subjects not gained during a three-years' course, or during the longest sojourn in a research laboratory. The industrial chemist may have the research instinct, and he will not need to starve it, but his hunger after new knowledge for its own sake must not prevent him from giving his interest and great enthusiasm to minute questions of economy, to the choice of the cheapest materials, to labour-saving devices, thermodynamic considerations, questions of fuel economy, and so forth. Such work demands not merely a type of ability as specialised as that of the research chemist, but, above all, a specialised type of training which it is our business now to consider.

In speaking of the technical or industrial chemist, I have in mind, perhaps, a more particular type of man than the actual expression implies, namely, the person who gets to grips with the actual chemical or industrial process carried out on the large scale; in this country the heavy chemical and metallurgical industries, as well as those concerned with the production of fats, oils, soaps, starch, sugar, tanning, etc., have on the whole been moderately well developed, and have in many respects even excelled. In the synthetic organic chemical industries, on the other hand, the elaboration of residuals, the manufacture of intermediates, dyes, photographic chemicals, drugs and the like, have not, prior to the war, succeeded, and we have been outstripped by foreign competitors. I think we may find in this distinction some indication as to the line we should pursue in education in order to remedy the latter condition. In the organic chemical industries there is a great diversity of operations involving expert knowledge; frequently the demand for the product concerned is on such a scale that its manufacture cannot be carried on continuously all the year round, so that the diversity of functions required for individual manufactures—much more complicated than in the case of the first-mentioned class of substances—is still further extended by the making of now one compound and now quite a different one. The operations require a very close

\* Paper read before the Old Students' Association of the Royal College of Science, Feb. 8, 1921.

attention to details, and quite small variations of conditions may entail great reduction of yield and result in financial losses. Now, those responsible for the supervision of such work must not only be skilled, but highly educated men of a particular type who, when produced in this country, hitherto have been self-trained and not the direct product of the university; but I suggest that it is not only possible, but of urgent importance that we should give a suitable training for this purpose. It is true that the right men are few, and it therefore takes a big net to catch them. It would surely be of immense advantage if students admitted to any such post-graduate course in a college were graduates widely drawn from the various universities of the country. Such moving about might with great advantage be encouraged more than it is.

Chemists in the heavier industries referred to above can in a large measure carry on their work from the laboratory and the office, giving instructions to the foreman whose rule-of-thumb knowledge constitutes a very real bulwark, but in the manufacture of fine chemicals processes need to be directed by a chemist on the spot. This chemist must be a man who inspires confidence, and can act with courage born of conviction. A man who not only knows, but who knows that he knows, and, above all, a man willing to don overalls and to dignify his works and himself by practical co-operation on the plant.

If you will take the view that for such positions men can, by training, become better fitted for the work earlier in life, what is the nature of the training required? A sound fundamental knowledge of chemistry, physics and mathematics is a condition of graduation; super-added to this must be a not inconsiderable training in engineering, machine construction and machine drawing; together with knowledge of fuel economy, chemical kinetics, thermochemistry and electrochemistry, works management, costing and accountancy, and chemical economics. By the last-mentioned, I mean a study of the efficiency of established processes, *e.g.*, alkali, acid and explosives manufacture, and nitrogen fixation from a point of view well illustrated in the Government publications relating to the achievements in war factories during the war. The historical review of these processes would constitute an admirable basis for a course of study. These may be treated as classroom subjects, but they cannot be effectively studied without very special practical training in a technical-chemical laboratory. Such a laboratory would preferably be nothing less than a manufacturing undertaking, but it would not deal with specific manufactures, such as sulphuric acid or soap or brewing or dyeing, as these may be regarded as the function of trade schools which are able to undertake useful teaching of a different kind.

I wish particularly to emphasise the point that there are special underlying principles relating to the application of chemical science to manufacture which can be taught and be of excellent educational value. It may be argued that a one-year's curriculum is all that is absolutely essential, but to this I am not personally inclined to agree; however, the courses might conceivably be so arranged as to leave the way open for some students to break off at the end of the first year, while others spend a second year at laboratory work. I believe, however, that a second year would prove of inestimable value and be readily adopted by students.

In addition to the general classroom subjects referred to, I would make provision for courses of lectures and reading on groups of chemical subjects somewhat as follows:—

(1) Dyes and dyestuffs, synthetic drugs, essential oils, sugars, alkaloids, biochemistry;

(2) Coal tar, petroleum, fatty oils, alkali, and acid;

(3) Colloid chemistry, rubber, tanning, cellulose; students being expected to take at least one of these courses.

The success of all attempts to teach chemical technology will depend, however, chiefly upon the opportunity to put knowledge into practice in the laboratory, to develop initiative, resource, and self-reliance in dealing with processes, and in translating laboratory experiments to works-scale manufacture. Often does it happen that a chemist believes a foreman's method to be capable of improvement, yet he has not the varied experience which will justify him in interference, and thus his initiative is discouraged and suppressed. It must be remembered that the majority of foremen secretly resent improvements introduced by another, and will even resort to trickery to render unsuccessful any modification of process suggested by a chemist.

Now, manufacture cannot well be carried on in the laboratory unless the goods manufactured are going to be made use of in one way or another. There are both psychological and practical reasons for this. Materials must be used, and the cost of them, unless the finished product be sold, is a serious deterrent to free experiment; further, a student is not satisfied to expend care on producing a substance which will be wasted and will not come before the tribunal of public criticism; nor would it be good for him if he could be induced to work regardless of these factors.

The scheme advocated consequently involves the formation of a technological teaching laboratory of a new type, one which would be a miniature manufacturing establishment associated with a college providing the necessary lectures and class-room instruction.

In addition to chemical manufacture, technical-scale experiments extending the results of investigations in research institutions or elsewhere might well be undertaken; one would expect that there are enough small demands for compounds not supplied in the ordinary way of trade, as well as for these technical scale experiments, to keep such a college laboratory perpetually engaged in useful work. The compounds manufactured would include such of the less common compounds of rare application as the manufacturer will not be troubled to supply, because the demand is too small and irregular. This manufacturing laboratory would need a permanent staff of experienced men who would act as demonstrators and take a personal part in the practical work. The laboratory would need to be run under conditions of strict and complete business organisation and discipline, with long hours of attendance. Powers practically equivalent to that of dismissal would be entrusted to those in control, and no student would be permitted to proceed from one stage in his course to another without having qualified in the earlier one.

1. Structurally the laboratories would consist preferably of a lofty shed about 27 ft. high, of steel construction, well lit and ventilated, the steel being arranged so that staging may be erected at 9 ft. and 18 ft. from the floor, in which technical-scale plant can be rapidly erected. The successful use of this laboratory would in large measure depend upon the provision of standardised pans, covers, columns, and stills, which can be rapidly fixed in position, such engineering work being chiefly carried out by students.

This laboratory might well contain a few special plants designed for typical manufactures suitable for determining efficiency on short runs, such, for instance, as nitro-toluene, formaldehyde, phosgene, ammonia oxidation, benzoic and salicylic acid, the products from which would be utilised for further processes.

2. A large kitchen laboratory fitted with small apparatus of 50 to 100 litres capacity, including typical centrifugal filtration and evaporation plant,

autoclaves, hydraulic press, and so forth. This building should also preferably be a single storey building, and contain a good laboratory for investigations of a preliminary character.

3. The drawing office, library and administrative offices, stores, and the analytical and physical laboratories, each separate rooms, might be included under one roof.

4. The workshops—wood and iron—foundry and forge, and plumbing shop would constitute another departmental group of buildings.

5. A power plant, generating steam, gas, and electricity under conditions which enable a daily computation to be made of the efficiency of each is an essential adjunct. The steam, gas, and electricity would be distributed to separate sections under a scheme of meters to enable the various operations to be accurately assessed with the consumption.

The fundamental object of the instruction would be to introduce the cost quantity in relation to power, heat, labour, cost of material, and yield of product. The observance of these principles concerns the chemical industry more especially than any other, because in that industry costs may vary over an enormous range according to the care and skill exercised. To assist the student in this attitude I consider instruction in practical accountancy, costing, and stock-keeping to be of fundamental and first importance. The care, initiative, and resource which need to be exercised, the practice in approaching problems on economic lines, and the practical knowledge gained, combined would constitute an admirable preparation for industrial management in many spheres, by no means confined to organic chemicals.

During the first year the student would attend courses of instruction in physical and electro-chemistry, construction, and design, chemical engineering, and machine drawing. He would also receive practical instruction in the workshops, power-house, analytical laboratory, administrative office, and in the use of special machinery and plant. He would take his turn at stoking, engine driving, plant erection, lead-burning, the making of steam joints, and so forth.

Passing into the works laboratory, first-year students would at first manufacture simple compounds, and be given an opportunity of assisting the operations of advanced students. These manufactures would need to be conducted under strict discipline as regards both time spent and quantitative results, and although from its nature the work would not be on the full scale of ordinary technical procedure, it would be carried out on a sufficiently large scale to require manufacturing apparatus as distinguished from that of the laboratory. During the first and second year the student would become gradually familiar with the construction and use of autoclaves, large reaction vessels, vacuum pans, gas compressors, furnaces, centrifuges, and filter presses, and would acquire experience in handling difficult substances, in utilising catalysts, etc. He would also have experience in designing special pieces of plant for particular technical processes. The courses of lectures would include steam-raising and power production, every student spending several weeks in the department concerned with the supply of steam and power to the institution, and in a laboratory in which fuel, water and flue gases, etc. are analysed. Courses of instruction in special manufactures, such as sulphuric acid and alkali, as already stated, would probably find their places in the curriculum: arrangements might be made for students destined for particular industries to attend special courses of instruction given by men actually engaged in the industries in question—a proceeding which has already been adopted in the evening classes of some of our technical colleges.

Further, it would be an invaluable adjunct to such a college if arrangements could be made for students to attend courses of practical instruction in actual works. By careful arrangement this might be done with such slight interference to the business of the firms concerned during only a short period in each year, that the opposition to receiving such visitations would be satisfactorily met. Above all, it is essential that the staff of the college should consist mainly of men of ripened works experience—not, he it understood, men (however able) who have merely spent a period in the research or analytical department of a works. Such men have usually been regarded by our educational authorities as men with works experience, when in point of fact often they have little or no real claim to a knowledge of chemical manufacture.

As regards the nature of the substances manufactured, the first-year students might be occupied partly in making from the raw material simple inorganic and organic substances; for instance, potassium dichromate and permanganate, metallic arsenic and tellurium, hydrogen peroxide, hydriodic acid, ammonium persulphate, nitrations, sulphonations and the like organic operations, and partly in isolating and purifying tar products, sugars, and natural vegetable products, such as theobromine and caffeine.

In the second and succeeding years—for it may safely be assumed that many students would remain even longer than two years, and it should prove possible to pay them to do so—the students would be occupied chiefly in carrying out large-scale technical research; they would be enabled to follow each other's processes, being present to watch critical stages. The compounds manufactured would, in general, be those which, while not in common use, are required for exceptional purposes, chiefly by colleges and research laboratories. In order that the scale may be a large one it would be necessary to make of many substances much bigger quantities than are likely to be required; these might remain in storage for many years, and only gradually would a complete list of substances in demand be accumulated.

One claim for this scheme is that, besides affording a really practical training, it would maintain and impart by means of actual manufacture and sales a standard of industrial efficiency almost impossible of attainment without this incentive. It would also have the practical value of supplying many chemicals for which we were formerly dependent upon Germany. Nor need we fear that such a scheme would give serious offence in commercial quarters, for the monetary value of business in the manufacture of fine chemicals of this type is of no great account, and it would not be difficult to avoid jealousies by confining the manufacture to those substances which manufacturers of fine chemicals do not wish to produce, and by rigorously adhering to a scheme of distribution through existing trade channels. If a manufacturer should complain that he is subjected to unfair competition through the production of a certain chemical by an endowed and privileged institution, the reply is simple: Let him produce a supply of the substance of adequate purity, and it may be assumed that the college of my conception would leave it to him.

Incidentally, the college would prove of great advantage to research workers, who would have here facilities for procuring the compounds with which their investigation commences, instead of spending much of their valuable time in repeating tedious preparations on a small scale. This single benefit, increasing as it would the effectiveness of research workers, would react favourably on the industry as a whole.

It may be objected that the student cannot lengthen his course at college by a further period of two years. A medical student does not become

qualified in less than five years. Why should a chemical technologist expect to do so? I venture to think he would have to apply himself just as strenuously to achieve what is required of him in the time. The two years would be spent much more effectively from the point of view of increasing the student's monetary value than any other two years of his education, and so far as many are concerned, more profitably than if spent on research.

As to the financial aspect of the scheme, it may be agreed that as manufacturers would gain much they should be looked to for assistance. With chemical industry threatened as it is at this moment, one hardly dare look for much help in this quarter; nevertheless, one might confidently expect that when successfully established the fine chemical industry would afford a substantial contribution towards the maintenance, but the equipment must be costly and the buildings substantial. Surely for this we must look to the Government, whose duty it is to direct our educational organisation with a greater bias to practical ends. Enough to build and endow such a college many times over would have been saved during the war from the vast expenditure on chemical war factories had we had the foresight to establish the fine chemical industries here previously, and a dozen students from a college such as that which I have endeavoured to conjure to your minds might have saved millions. But these millions will only be truly lost if, failing to learn the lessons of the war, we allow our fine chemical industry to slide back to the condition in which it was when war commenced. We escaped disaster once; be sure we should not so escape again.

## A BELGIAN BUREAU OF CHEMICAL STANDARDS\*

*Scientific and Technical Importance of Very Pure Chemical Substances.*—It is well known that the purity of materials plays an important and often an indispensable part in many branches of science and technology. Thus the individuality of many chemical types can only be established by preparing very pure elements, and such work has led to the discovery of numerous elements (e.g., rare-earth elements, rare atmospheric gases) and of entirely new classes of compounds (thiophen an impurity in benzene). Certain physical properties can only be determined by employing pure substances (super-conductivity of metals in liquid helium), and others are inseparably associated with the presence of certain definite impurities (phosphorescence). The progress of stoichiometry depends above all on the investigation of the constants of chemically pure substances. In metrology there is a tendency to replace the use of standardised instruments (metro scale, standard thermometer, etc.) by the adoption of selected constants of well-defined substances. The use of very pure products has also become equally indispensable in technical work, for example, for analytical reagents and the fundamental constants of alcoholometry (density of absolute alcohol); electrolytic copper; pure gases in large-scale catalytic processes; and in biology and medicine, the use of very pure chloroform as an anæsthetic, and of very pure sugars for certain bacteriological cultures.

*How can they be obtained?*—Both chemists and physicists are often held up in their work by difficulties in obtaining specimens of very pure materials essential to their investigations. Except in special cases, sufficiently pure substances cannot be purchased, whilst those obtainable have to be purified and their purity established. But the purification of small quantities of substances is often very difficult and the criteria of purity are not sufficiently sensitive; the preparation of larger quantities would save much trouble, e.g., in the case of mixtures of isomers or homologues. All physical chemists know, for example, the great amount of work entailed by every worker having to prepare such a substance as pure normal pentane. The war, finally, has much increased these difficulties by diminishing the availability of materials required in the preparation of pure substances.

Such considerations as these have been occupying the minds of chemists in the United States and Germany, even during the war, and have led them to take steps to fill in the *lacunæ* and thus make their work easier, more expeditious and more fruitful.

*What has been done in the United States and in Germany.*—The Physikalisch-Technisches Reichsanstalt at Charlottenburg has undertaken to make "normal" metals available to German chemists. Research has shown that metals sold as "pure" frequently contain more than 1 per cent. impurity; that it is impossible to prepare on an industrial scale, and without undue trouble, specimens containing less than 0.01 per cent. impurity; and that such substances—classed as grade 4†—suffice for the determination of atomic weights. The Reichsanstalt has therefore initiated a series of researches with the object of determining industrial processes of purification and methods of analytical control. Once these have been worked out, the preparation is entrusted to an industrial firm under the control of the Reichsanstalt; and the specimens are sold with a guarantee of purity certified by it. Similar arrangements have been made with regard to mercurous sulphate (for the Weston cell).

In the United States an analogous task has been undertaken by the Bureau of Standards at Washington, which prepares and sells specimens of alloys and minerals of known composition for analytical control, pure metals for thermometric standards, and certain organic compounds of which the method of purification is minutely described; naphthalene and benzoic acid for calorimetric standards, ammonium oxalate and dextrose for analytical work, and cane sugar for polarimetry.

American chemists, on their side, have created an industry of pure organic products in order to be independent of Germany. The Pfanzstiel Company, for example, specialises in bacteriological sugars, and the Kodak Company either produces or distributes gratuitously more than 750 organic compounds. All these efforts are co-ordinated by the National Research Council, and the work already accomplished is very remarkable.

*Plans of the Belgian Chemical Society.*—The above examples have instigated the Société chimique de Belgique to undertake similar work by providing a collection of pure standard substances, to be located partly in the University of Brussels. At the conference of the International Union of Pure and Applied Chemistry held in Rome (July, 1920), the Belgian delegates MM. Lucien and Swarts presented a memorandum by M. Crismer detailing the reasons for establishing such a collec-

\* Translation, slightly abridged, of a paper presented by J. Timmermans to the Société Chimique de Belgique on January 30, 1921, and published in the Bulletin de la Société Chimique de Belgique, Vol. 30, 1921:

† The Reichsanstalt prescribes five grades of impurity as follows:—Grade 1, containing from 1 to 10% of impurity; grade 2, from 0.1 to 1%; grade 3, from 0.01 to 0.1%; grade 4, from 0.001 to 0.01%; and grade 5, from 0.0001 to 0.001%.



tion. In response, the conference decided to establish a Bureau of Chemical Standards and to entrust its organisation to Belgium. Already before the war the author had started a collection of pure organic liquids, and thanks to the help of M. Chavanne had received a grant from the Solvay International Chemical Institute which enabled him to collect a number of organic products to serve as starting material for the preparation of standards. The programme has been sketched out as follows:—

(1) It is proposed gradually to extend the existing collection until it contains samples of every well-defined chemical product, but in the first place efforts will be concentrated on, although not confined to, the collection of pure organic liquids, substances which are particularly difficult to obtain at the present time. The collection should therefore contain representatives of typical organic substances which can be preserved in a state of purity without too much difficulty. For this reason it is not proposed to keep esters which are readily hydrolysed, but only the acids and alcohols from which they are derived.

(2) It is intended to maintain a depot of standard substances which have been prepared in the United States, or in any country adhering to the International Union, so that the results of the labours of American chemists may be made available to their colleagues in Europe.

(3) Small quantities of the standard substances will be placed at the disposal of Belgian and foreign scientists to save them the tedious work of re-purification. It will therefore be desirable that the samples shall be made from single batches of materials weighing several kilograms; and this will inevitably lock up considerable capital.

(4) The Bureau will act as a centre of information on all matters appertaining to pure products, rare specimens, and the relevant literature.

*How to help the Bureau.*—(1) To-day more than ever money is the nerve of action, and therefore all who appreciate the importance of the work are asked to aid in developing its financial resources. Such an undertaking far exceeds the power of any one man, but a relatively small endowment would suffice for the institution of research scholarships to enable the Bureau to obtain the necessary collaborators.

(2) Industrial firms will be free to make a substance once the method of preparation has been definitely established. The Bureau, content with its rôle of pioneer, will then be able to turn its attention to new preparations. It is hoped that the example set by the Kodak Company in America will be followed in Europe. Manufacturers, in their turn, will be able to render great assistance by supplying the necessary starting materials and raw products, so reducing the amount of capital locked up in the collection; in this way they would encourage scientific work of general interest.

(3) In conclusion, directors of laboratories can greatly assist their colleagues by placing at the disposal of the Bureau specimens of pure materials or rare substances which they have made in the course of their work and which may be useful as standards. If a director wished to retain the material himself, he could make known to others, by means of the Bureau, that his specimens were available; and exchanges could be effected that would be advantageous to all concerned. For a similar reason specialists in this sphere are asked to keep the Bureau informed of the progress of their work, in order that it may be made known to all interested in the subject.

With the help of Belgian and foreign chemists it is hoped to carry out this programme of work; and for this the memory of Stas, that pioneer of chemical standards, will act as a stimulus.

## TURPENTINE IN THE UNITED STATES.

A recent bulletin by F. P. Veitch and V. E. Grottsch (U.S. Dept. Agric., Bureau of Chem. Bulletin No. 898. 1920) contains much useful information on the production, marketing, and uses of turpentine in America. About 75 per cent. of the world's supply of turpentine is produced in the United States, chiefly from the southern longleaf yellow pine (*Pinus palustris*), but other species, such as the Cuban or slash pine (*P. caribaea* or *heterophylla*), the rosemary pine (a variety of the loblolly pine *P. taeda*) and the western yellow pine (*P. ponderosa*) are also tapped; most of the remaining 25 per cent. is obtained in the south-western coast regions of France from the maritime or cluster pine (*P. pinaster* or *maritima*). The relative production of turpentine in the various States of America is approximately as follows:—Florida, 37 per cent.; Georgia, 19 per cent.; Louisiana, 15 per cent.; Alabama, 12 per cent.; Mississippi, 9 per cent.; Texas, 7 per cent.; North and South Carolina, less than 1 per cent. Formerly South Carolina produced the bulk of the world's supply of turpentine. At present less than 10 per cent. of the total is wood turpentine obtained from stumps and deal or fallen timber by steam or destructive distillation; this source of turpentine is becoming more important.

The oleo-resin is obtained by scarifying or "chipping" the living trees. A V-shaped cut is made about once a week immediately above the last cut, thereby forming a "face," down which the gum exuding from the cut flows and collects in galvanised iron or baked clay cups holding 1 to 2 quarts. The tapping season lasts from early spring to late autumn, the oleo-resin being removed from the cups about once a month and taken to the still in barrels.

The distillation plant is simple and has not been improved materially during the last 50 to 60 years; it consists of a copper still of 500 to 1000 gallons capacity, with a still-head (generally removable) connected to a large copper condensing-coil cooled in water. From 7 to 14 barrels of oleo-resin go to make a charge, depending on the size of the still and the nature of the oleo-resin.

The oleo-resin contains some water, and the distillate at the commencement consists of about 45 per cent. of turpentine and 55 per cent. of water. When most of the water originally present has passed over, as shown by a decrease of the water contained in the distillate to about 30 per cent., and by the peculiar sound which is heard close to the tail-pipe of the condenser coil, a small stream of water is admitted to the still. The distillate is collected in a barrel in which it separates from water; it is desirable to cover the barrel to avoid loss by evaporation. The yield of turpentine varies from 16 to 22 per cent. The turpentine is mostly shipped in wooden casks holding 50 to 53 U.S. gallons (231 cb. in.). These barrels are treated internally with two coats of glue to prevent the turpentine from penetrating the wood; steel drums are used only to a slight extent, but the larger producers, especially in the more western States, employ tank-cars of 5 to 10 thousand gallons capacity. On arrival at the primary markets each barrel or tank-car is inspected, and the grade (colour), purity and freedom from water, and volume of the turpentine determined.

The total production in the U.S.A. for the 1919 season amounted to 366,000 casks (50 galls.) of turpentine and 1,237,000 barrels (500 lb.) of rosin; during 1920 up to August 1, 237,155 casks of turpentine and 712,387 barrels of rosin were produced, the estimated production for the rest of the season being

233,876 casks of turpentine and 739,679 barrels of rosin.

The bulletin gives details of the market customs prevailing in the U.S. turpentine trade, of the methods employed in gauging barrels and tank-cars, together with references to the laws governing the sale of turpentine in various States, and also quotes the specifications recommended for turpentine by the U.S. Inter-departmental Committee on Paint Specification Standardisation (Oct., 1919), with full details of the methods of sampling and analysis.

## SOCIETY OF CHEMICAL INDUSTRY.

### ANNUAL MEETING, 1921.

#### *Accommodation on the s.s. "Melita."*

Members and their friends who intend proceeding to Canada for the meeting, and who have decided to travel with the President and his party on the "Melita" (not the "Megantic," as inadvertently stated in the last issue), are requested to secure their berths with the least possible delay. The offices of the C.P.O.S. are: Pierhead, Liverpool; 62-65, Charing Cross, Trafalgar Square, S.W. 1; and 103, Leadenhall Street, E.C. 3; at any one of which arrangements may be made.

#### *Return Sailings from Montreal.*

For the information of those who desire to return *via* Montreal, the following sailings of C.P.O.S. steamers are given:—

	First Class	Second Class
	Minimum	Minimum
	£ s.	£ s.
Sept. 10. Empress of Britain .. ..	51 10	31 10
Sept. 16. Victorian .. ..	—	30 0
Sept. 20. Metagama .. ..	—	32 10
Sept. 23. Melita .. ..	—	32 10
Sept. 24. Empress of France .. ..	51 10	31 10
Sept. 30. Minnedosa .. ..	—	32 10

"Empress" steamers sail from Quebec only, but free railway tickets will be provided between Montreal and Quebec. All other steamers carry "cabin" class only from Montreal. Members and their friends who wish to return home by one of these boats should communicate with the C.P.O.S. before the end of June, and the company will arrange for reserved accommodation.

#### *Passport Regulations.*

The following information regarding passport regulations should be noted: Passengers traveling to the United States *via* Canada and passengers proceeding to Canada *via* the United States must obtain the U.S. consular *visa* on their passports before they leave the United Kingdom. Passengers who are not U.S. citizens must make *personal application* for *visa* at the U.S. Consulate nearest their place of residence. One photograph of each passenger is required. Application should be made not less than two weeks prior to the date of sailing. Passengers travelling from the United Kingdom to the U.S. *via* Canada cannot obtain the American consular *visa* in Canada; it must be obtained in the United Kingdom prior to sailing. Forms and full information regarding procedure are obtainable at the Canadian Pacific offices.

**U.S. Head Tax.**—As required by the U.S. Government, all passengers (excepting U.S. citizens) proceeding to points in the United States must pay at the time of booking the head tax of £2 5s. per person in addition to the ocean fare; children under 16 years of age accompanying their father or their mother are exempt.

## VISIT TO SHAWINIGAN.

Arrangements have been made to take the visiting members of the Society to Shawinigan Falls immediately after the conclusion of the Annual Meeting. A special train is being provided for members and their friends, which will leave Montreal at 11.30 p.m. and arrive at Shawinigan Falls next morning.

Shawinigan Falls owes its existence to the splendid electric-power development; prior to 1898 it was a forest wilderness, but the year following the Shawinigan Water and Power Co. installed two 5000 h.p. generators, which formed the nucleus of the present development of the district, now amounting to over 300,000 h.p. It is estimated that the district has a potential capacity of over a million h.p.; this power is the foundation of a group of chemical industries which compare favourably with any on the American continent using over 130,000 h.p.

The pioneer chemical industry to be established was the Northern Aluminum Co., which started operations about 1901 and now uses ten times as many h.p. as at that time. The principal raw material—bauxite—has not yet been discovered in Canada, but is imported from Arkansas. The process followed is the well-known Hall method of producing aluminium by direct current, using a bath of alumina in fused cryolite. In addition to manufacturing aluminium, the company also maintains a wire-drawing plant, and makes all the aluminium wires and cables used in Canada.

Following closely on the heels of the Aluminum Company, the Belgo-Canadian Pulp and Paper Co. started operations in 1903. This undertaking owns large timber areas on the St. Maurice River and its branches, down the streams of which the logs are floated to the mill. The company produces both ground-wood and chemical pulp, and in addition makes over 90 tons of paper per day.

The Canada Carbide Co. was established in 1903, the first installation being one small single-phase electric furnace. From this small beginning it has grown to such an extent that it now occupies some fifteen acres of land, upon which have been erected lime-kilns, buildings for storage, two main rooms where the carbide is produced in furnaces ranging from 3000 to 10,000 h.p. The carbide is crushed, sorted and packed in separate rooms, and there is a small plant for manufacturing steel drums and wooden crates for shipping.

The Canadian Electro-Products Co. came into existence to supply the demand for acetone in cordite manufacture during the war. Production from calcium acetate obtained by wood distillation proved entirely inadequate and, at the request of the British Ministry of Munitions, the Shawinigan company started investigating the possibility of producing acetone synthetically from acetylene. The experimental work was begun in December, 1915, and after a half-year's intensive research by five Canadian chemists, construction was commenced in May, 1916. By January, 1917, the whole plant, covering fifteen acres, was complete, and the first car of acetone shipped to England. The working out of the process, of which the steps are carbide+acetylene→acetaldehyde+acetic acid→acetone, involved the development on a large scale of reactions hitherto only performed in the laboratory and which presented difficult and unusual problems; and it is very much to the credit of those concerned that when the plant was doubled at the entry of the United States into the war only a few minor changes were made in the design and arrangement of the equipment. The production exceeded 10,000 tons of acetic acid in 1918. Acetone is not manufactured at the present time; the process is stopped at the acetic-acid stage, 99 per cent. acetic acid being obtained in one distillation.

Plans are now being made to produce acetic anhydride, monochloroacetic acid, aspirin, etc.

The Canadian Aloxite Co., a subsidiary of the Carborundum Co. of America, started about two years ago and is gradually increasing its operations, so that in the near future it will utilize 20,000 h.-p. The products are ferro-silicon and abrasives.

The demand for electrodes for the electric furnaces at Shawinigan Falls and Montreal led to the formation of the Canadian Electrode Co., which has a plant capable of meeting all the requirements of the locality. Only carbon electrodes are now made.

Rarely can one find in so restricted an area so many industries of which the life depends upon chemical research.

#### MEETING OF LEATHER CHEMISTS IN NEW YORK.

A letter has been received from Mr. J. A. Wilson, chairman of the Leather Chemistry Section of the American Chemical Society, extending a very cordial invitation to the leather chemists of this country to attend the sectional meetings in New York City during the week beginning September 5, 1921. It is anticipated that some very important papers will be read which will mark the beginning of a new and vigorous development of the chemistry and microscopy of leather manufacture. Papers in course of preparation relate to the microscopy of hide and leather at all stages of the process, the action of enzymes in bating, the chemistry of the tannins, chrome and vegetable tanning, and vegetable tannin analysis. British chemists are invited to contribute papers, and if they cannot attend the meeting they are asked to send them to Mr. Wilson (203, Juneau Avenue, New York) to reach him not later than August 6.

#### NEWS FROM THE SECTIONS.

##### NEWCASTLE.

A dinner was given by members of the Section on April 23, in the Central Station Hotel, in honour of the President of the Society. Dr. J. H. Paterson presided, and among those present were the Rt. Hon. the Lord Mayor of Newcastle, Mr. A. E. Doxford (chairman, N.E. Coast Institution of Engineers and Shipbuilders), Mr. G. Vardy (president, British Foundrymen's Association), Dr. J. P. Longstaff, Professors P. P. Bedson, Henry Louis and W. N. Haworth, Dr. J. T. Dunn, and Mr. E. Morley Fletcher.

Dr. Paterson, in proposing the toast of the Society, coupled with the name of the President, said that they were all proud of the Society, which was one of the most influential societies in the country, if not the largest numerically. They were proud of the Society's *Journal*, which was one of the most advanced and best-arranged journals in the world, and they were also proud of their President, whose work was known and appreciated far beyond the ranks of their own members. Sir William Pope said that he was fully confident that the future of the Society would be as glorious as its past. The criticisms that were occasionally heard really demonstrated that the Society was alive, and that progress was being made. He agreed with the chairman that the *Journal* was one of the foremost in the world. After alluding to the great part which the Newcastle Section had played in the success of the Society, and to its distinguished past-presidents, Sir William Pope spoke of the forthcoming annual meeting in Canada, and appealed to the Section, and to all the other Sections, to send as many members as possible to take part in it.

Prof. P. Phillips Bedson proposed the health of the Lord Mayor of Newcastle, Prof. H. Louis that

of the guests, Dr. J. P. Longstaff that of Armstrong College, and Dr. F. C. Garrett that of the hon. local secretary and hon. treasurer. The chairman gave the health of Prof. Bedson, who has been a tower of strength to the Newcastle Section, and who is shortly to retire from the chair of chemistry at Armstrong College, after nearly 40 years' service.

At the annual meeting which preceded the dinner, the officers of the Section were re-elected, and the following were elected members of the committee, out of 18 nominations:—Messrs. P. E. Bowles, W. Diamond, J. T. Dunn, F. C. Garrett, F. Hirsch, D. W. Jones, H. Louis, H. Peile, A. Short, F. H. Walker, and G. Weyman.

##### BIRMINGHAM.

The annual meeting was held in the University buildings on April 14, Dr. H. W. Brownson presiding. There are no changes to report in the list of officers of the Section, but the following were elected to fill vacancies on the committee:—Messrs. W. Brazier, G. H. Blenkarn, F. L. W. Bradford, H. T. Pinnock, and W. C. Davis.

Dr. E. B. Maxted read a paper on "The Manufacture of Pure Hydrogen and the Catalytic Hydrogenation of Oils." In discussing the four methods by which hydrogen is now usually prepared for use in industrial catalytic processes, the author stated that the electrolytic and the cyclic water-gas processes gave sufficiently pure hydrogen for direct use, but the gas obtained by the low-temperature separation of the constituents of water gas and by the catalytic water-gas process required to be further purified from carbon monoxide. The cyclic water-gas method was particularly suitable for manufacturing hydrogen for the hardening of oils. Dr. Maxted then explained the origin of the small amount of carbon monoxide occurring in the hydrogen produced in the last-mentioned process, and pointed out that the deposition of carbon, which was the *bête noire* of this process, could be easily prevented by varying the composition of the gas used for reduction, viz., by using in place of ordinary water gas a gas somewhat richer in carbon dioxide, such as was obtained with a water-gas producer constructed for operating at an abnormally low temperature. In the hydrogenation of oils, it appeared that traces of oxygen or nitrogen acted principally as diluents, but in certain other catalytic reactions, traces of oxygen acted as an inhibitor, and it was then necessary to use water for generating the steam which was as free as possible from dissolved air. The *modus operandi* of hydrogenation was then outlined, and this was followed by remarks upon the nature and activity of the nickel catalyst, the influence of temperature and speed of reaction. In the hydrogenation of ordinary commercial oils, it was the author's practice to begin hardening at 120°–130° C. and to allow the temperature to rise to a maximum of about 160° C.

##### GLASGOW.

A paper on "Tar Distillation" was read by Mr. W. A. Wahmsley, of the Glasgow Corporation Chemical Works, at a meeting held on March 22, Mr. J. H. Young presiding.

Crude tar is difficult to distil when any ammonia water remains in it; an actual charge of 30 tons in a pot still requires 22 hours for the removal of water and crude naphtha, and only 6 hours further to complete the distillation to pitch. Modern stills are designed to obviate this long period of distillation of water and naphtha, and at the same time to economise fuel. The vapours from the stills are passed through coils covered by crude tar in a

pre-heater, whereby they are cooled and the crude tar is dehydrated. A further development of this principle is found in the continuous tar-distillation plant. Incoming tar is gradually warmed by meeting outgoing vapours, and in passing through a series of stills is stripped of fraction after fraction until pitch only is left. The system is not elastic enough in its present form.

Dehydration by pumping crude tar through heated coils and releasing suddenly to atmospheric pressure is being successfully accomplished, and the same system is used in working up crude oils to recover naphtha, this product being liberated on expansion.

The manufacture of caustic soda for phenol recovery is an essential process, and for its preparation on a large scale a continuous system is adopted. The settling of precipitated calcium carbonate is accelerated by gently moving paddles, and the sludge is continuously dried on a revolving filter ready for recalcination, if this is commercially justifiable. Little power is used in mixing the oils with caustic soda solution when the agitation is done by creating a vortex and breaking the surface by shallow baffles.

In rectifying naphthas, a very good column is one in which the vapours travel in a downward direction in each section and are subjected to the scrubbing action of descending condensate.

The entire works and individual plants should be designed to provide a continuous flow of products in one direction, effluents also tending in one direction.

The above processes are in operation or are being installed in the Chemical Works Department of the Glasgow Corporation.

#### LIVERPOOL.

The annual meeting of the Section was held on April 29 at the Adelphi Hotel, Liverpool, Dr. E. F. Armstrong presiding. Dr. G. C. Clayton, of the United Alkali Co., was elected chairman, in succession to Dr. Armstrong, who now becomes vice-chairman. After many years' good service, Dr. A. Holt retires from the hon. secretaryship in favour of Mr. E. Gabriel Jones, but remains on the committee as hon. treasurer, Mr. Edwin Thompson becoming the Sectional representative on the Chemical Engineering Group. The following were elected to the committee in place of retiring members:—Messrs. C. O. Bannister, G. W. Beaumont, A. E. Findley, E. L. Peck, and Edwin Thompson.

After the business meeting, a paper on "Mustard Gas" was read by the President, Sir William Pope.

#### YORKSHIRE.

Mr. S. H. Davies presided at the annual meeting of the Section held at Leeds on May 2. The officers were re-elected, and as five members of the committee retired and one member had resigned, the following were elected to fill the vacancies:—Messrs. C. P. Finn, W. B. Hill, H. J. Hodsman, P. E. King, F. W. Richardson, and B. Watmough. The chairman said that unfortunately there had been few papers read during the past session, but it was hoped to have more next year, and it was proposed to hold meetings in centres other than Leeds, such as Sheffield, Huddersfield, or Bradford.

Prof. A. Smithells then gave a demonstration, with models, of modern views of the chemical atom, his aim being to set his audience re-studying the broad fundamentals of the subject, especially Dr. Irving Langmuir's paper on the arrangement of the electrons in the atom. The advance of chemical theory since Dalton's time was sketched, the conceptions of Dalton, Berzelius, and Van't Hoff being illustrated by models representing washing soda.

Study of radio-active disintegration pointed to the necessity of going beyond the atom and of postulating its complexity and an electrical theory of matter. The existence of the electron is now questioned by no one. The lecturer referred to the isolation of the negative electron and the association of mass with the positive electron, the Bohr hydrogen atom, and the Rutherford atom. Before describing the Langmuir atom, the postulate was put forward that in order to be of service to the chemist it must explain the chemical properties of the elements as expressed, say, in the periodic classification of the elements, and their stability chemically and physically must be in accordance with the theory. The Langmuir atom was then illustrated by models and its accordance with the properties of the elements of the helium series was brought out. Langmuir's view of the combination of the elements was demonstrated, and a model showing the structure of the sodium carbonate molecule was built up from models of the constituent atoms. Other interesting models dealt with the methane molecule, the similarity of the nitrogen and carbon monoxide models from the point of view of their inactivity, and the atom of nickel.

#### LONDON.

The annual meeting was held on May 2, at Burlington House, W. Mr. Julian L. Baker, the chairman, in the course of an address, announced that Mr. E. V. Evans was to succeed him in the chair, and that Dr. Monier-Williams had been re-elected hon. local secretary. At the six meetings of the Section which had been held so far eight papers had been read, and the average attendance had been 97 members and 21 visitors. The committee had had under consideration the question of holding joint meetings with the Chemical Engineering Group, and it was hoped that it would be possible to arrange occasional joint meetings of the Société de Chimie Industrielle and either the parent Society or the London Section. He appealed to members to make every effort to attend the Annual Meeting in Canada, and he also drew attention to the informal dinners of the Section which were held before the meetings, and at which a better attendance was desirable.

The following were elected to fill vacancies on the committee:—Messrs. R. T. Colgate, H. G. Colman, H. F. E. Hulton, S. Miall, H. P. Stevens, and E. W. Voelcker.

The following papers were then read:—(1) "Fractional Distillation with Contract-ring Still-heads," by Dr. R. Lessing; and (2) "Thermal Losses in the Gas Producer Process," by Mr. N. E. Rambush.

(1) Dr. Lessing's contact-rings consist of metal cylinders of approximately equal height and diameter, provided with a narrow slit from end to end and a diametrical partition connected to one side of the slit. They are dropped promiscuously into a cylindrical still-head, and owing to the large amount of free space remaining, about 87 per cent., the resistance in the still-head is almost negligible. The author's experiments have shown that the efficiency of these heads is greater than that of any other dephlegmator, and is due to the large surface per unit of volume, the turbulence caused by frequent deflection of currents of vapours and condensate, and the consequent absence of channelling. Maximum efficiency may be obtained by lagging or heating the still-head and by super-imposing a reflux condenser. The rings, which are made of copper or aluminium, are used in scrubbing-towers, condensers, heat-exchangers, etc., especially when a minimum of back pressure is essential: for such purposes the size recommended is 1 x 1 in., but smaller sizes are available for laboratory use.

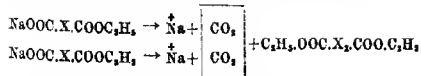
(2) Mr. Rambush's paper described the results of an exhaustive research on the cause of the heat-losses in the gas-producer process. The factors concerned in the losses are:—(a) The moisture-content of the fuel, which is roughly proportional to the thermal loss; (b) the amount of condensable volatile matter obtained by gasifying the fuel; (c) the loss of dust and soot due to high gas velocities and high temperatures, which may lead to a loss of 3 per cent. in the heating value of the fuel; (d) the ash content of the fuel—the higher the ash content the greater the amount of unburnt carbon; (e) radiation and convection, which cause a loss of about 1 per cent. in the heating value of the fuel gasified; (f) leakage due to poking, cleaning, and bad joints, which may amount to a loss of at least 1 per cent. of the heating value; (g) the mechanical removal of gas and the solution of gas and tar in the cleaning plant may cause a loss of 1 per cent.; (h) the water-vapour content of the gas derived from the undecomposed blast steam, the amount of which depends upon the time factor; (i) the heat carried away in the hot gases from the producer causes the highest thermal loss in cold gas producers, but is negligible in the case of hot-gas plant. When using fuel unevenly graded or in large pieces the fuel depth must be greater to ensure efficient gasification. The heat loss in the hot ashes is negligible, ranging from 0.1 to 0.5 per cent.

The author described his method of calculating the thermal efficiency of a producer plant, and his results were in good agreement with those obtained in actual practice. The relative losses obtained in various forms of plant when using an average English coal were found to be:—Hot gas producer (if cooled), 17 per cent.; cold gas producer (without recovery), 14 per cent.; and the ordinary Mond type of by-product producer, 16 per cent.

## MEETINGS OF OTHER SOCIETIES.

### THE ROYAL INSTITUTION.

The Friday Evening Discourse on April 22 was delivered by Sir James Walker on "The Electro-synthesis of Organic Compounds." The ionic theory of electrolytic conduction was briefly reviewed, and incidentally it was pointed out that electrolytic vessels now used are still very similar in form to those employed by Faraday, whose studies were confined entirely to the electrolysis of solutions of inorganic substances. In preparing ethane by the electrolysis of an aqueous solution of sodium acetate in 1849, Kilbe established his right to be regarded as the pioneer of the electro-synthesis of organic substances. In 1890 Crum Brown attempted to extend Kolbe's method by electrolysis sodium ethyl tartrate, but without result. The lecturer had then pursued the subject by attempting the electrolysis of sodium ethyl malonate. The result indicated a general method for the electro-synthesis of organic substances which may be represented by the equation:—



where X represents any organic group. The  $\text{C}_2\text{H}_5$ -group in the resulting synthetic product may be substituted by sodium and the synthesis carried a step further, whereby a product containing four X-groups is obtained. The method can be applied to the electrolysis of mixtures, thus enabling compounds to be synthesised which contain groups X in the order of their natural number. In the

particular case where X represents the  $\text{CH}_3$ -group, the electrolysis of sodium ethyl malonate results in the production of diethyl succinate. In this manner the lecturer had succeeded in synthesising from malonic acid, substances containing up to 16  $\text{CH}_3$ -groups. Substances containing 32  $\text{CH}_3$ -groups had not hitherto been synthesised in this manner—probably owing to electrolytic difficulties connected with the fact that these synthesised products were of the nature of soaps. The method had not hitherto been applied with success to the electro-synthesis of aromatic substances, but investigations were now being made. Generally speaking, investigation of the electro-synthesis of organic compounds had been much neglected in recent years.

### THE OPTICAL SOCIETY.

At a meeting held on April 14, Mr. F. Twyman, of Messrs. Adam Hilger, Ltd., described "An Interferometer for Testing Camera Lenses," which is an application to camera-lens testing of the principles of an earlier instrument described in the Philosophical Magazine (Phil. Mag., 35, Jan., 1918), but it differs from the lens interferometer there described in that the camera lenses can be tested for oblique pencils. The instrument gives aberrations in the form of an interference picture (that can be photographed), which is a contour map of the aberrations of wave-front expressed in wave-lengths of light in comparison with the wave-front of a perfect camera lens.

A paper on "The Testing of Heliograph Mirrors and the Measurement of Mirrors of Long Focal Lengths" was read by Mr. W. Shackleton. The method of testing the general surface consists in observing the reflection from the mirror of a system of squares on the test board, the magnification given by the mirror being utilised to determine the curvature. The method of examination is applicable to the rapid measurement of mirrors of long focal length, especially of those forms silvered on the back, cata-dioptric mirrors, where the usual methods of measurement, test planes or spherometers, are inapplicable. Defects of curvature and distortion, astigmatism, and strain in the glass can be detected, and also defects of silvering and had polishing.

### THE INSTITUTE OF PHYSICS.

The inaugural meeting of this new professional body was held in the hall of the Institution of Civil Engineers on April 27. Sir Richard Glazebrook, who presided, urged the claims of physics in national life, and said that its utility during the late war had done much to impress the nation with its essential importance. There were already about 300 members in the Institute, including most of the leaders in physical science.

Sir J. J. Thomson, the president-designate, compared the conditions to-day with those of 50 years ago. Then there were probably not more than about 100 physicists and a dozen small physical laboratories in the country; the only posts open to physicists were badly-paid professorships and worse-paid school appointments. The number of laboratories had very greatly increased, but the supply of physicists, although it had also increased, was far below the demand. There was a great need of physicists in industry, not only in the laboratories but in the works themselves. Science students at the Universities should spend at least one year on research, if only on account of the mental discipline it afforded. "Research develops character, increases independence of thought, and develops resourcefulness, critical power, and enthusiasm; in fact, it raises the student from intellectual ado-

lescence to intellectual manhood." The growth of physical research was illustrated by the increased number of original papers published; he estimated that there were about 2700 such papers in 1913, as against 400 in 1873. Although pioneer work was of the first importance, routine work was nevertheless essential.

The Rt. Hon. A. J. Balfour claimed that physics was the most fundamental of all the sciences, and said it was surprising that this country had had to wait until 1921 for the foundation of an Institute of Physics. He was doing his best to help on the admirable work of the Department of Scientific and Industrial Research, about which the public knew so little; but the public seldom knew much about what concerned it most. If half the time and half the money now being spent on petty squabbles about the division of the products of industry were devoted to increasing the power of man over Nature, there would be much more of those products to divide among the community. Expenditure of money and energy and the mere multiplication of students would not increase the number of great discoverers; but organisation and money were needed for that routine work which was absolutely essential to the promotion of great discoveries and to bring them to fruition. No money was better spent than on scientific research, and he was often surprised that the imagination of great magnates was not stimulated by the thought that by encouraging research they could effectively add to the wealth of the whole world. A fruitful discovery not only benefited the company, or capitalist, but it often touched the lives of every dweller in every civilised country, and no one could truthfully say that that was a low, material view of things. The idea of progress which held out most hope for the future was that the growth of science and invention would bring comfort and leisure—to those who knew how to use it—where at present discomfort and labour were the necessary conditions of production.

#### THE TEXTILE INSTITUTE.

The report presented to the annual meeting held in Manchester on April 27 refers to the loss by death during the year of Sir William Mather and of the president, Sir A. Herbert Dixon. The Foundation Fund, by means of which the Council hopes to embark on a definite programme of work, is as yet far short of the £50,000 required; nevertheless the annual income from the sum already raised has permitted an extension of activities which would otherwise have been impossible. The Compton Prize Scheme competition for advanced students is working successfully; the *Journal* has undergone important developments, and the institution of the annual "Mather" Lecture has proved of distinct value. There has been a substantial increase in membership, which now numbers 915.

In moving the adoption of the report the president, Colonel F. R. McConnel, made a number of suggestions for extending the scope and influence of the Institute, and said it was hoped that the committee charged with the work would soon complete the plans for a system of fellowships. The Institute, he said, was doing excellent service in showing how all ranks in industry could co-operate in attacking technical and scientific problems, and to that extent was influencing for good the relations between employer and employed.

After the business meeting Mr. W. E. Baker read a paper on "Ball and Roller Bearings as applied to Spinning, Weaving, and Finishing Machinery," which, from the chemical standpoint, was of interest, as it drew attention to the extreme cleanliness obtained by means of ball-bearings; at the present time large quantities of valuable material—

yarn, cloth, and liquors—are spoiled by the dirtiness of plain bearings and the drip and throwing of dirty oil from them.

#### SOCIETY OF PUBLIC ANALYSTS.

At the meeting held on May 4, Mr. A. Smetham in the chair, Messrs. F. G. H. Tate and J. W. Pooley contributed a paper on "Detection and Estimation of Illipe Nut Fat used as a Substitute for Cocoa Butter," in which they described a method of calculating the percentage composition of any mixture of these two fats from the densities, the melting-points of the fat and fatty acids, the viscosity and the iodine value. Dr. G. W. Monier Williams demonstrated an inexpensive apparatus for determining hydrogen-ion concentration, and discussed the underlying theory and its application to analytical work. In "A Note on the Oil of Oats," Mr. E. Paul described some work on the extraction with petroleum ether of whole-ground oats of the "Black Tartary" variety. Lecithins were found in the extract, and the constants of the separated oil were determined. The last paper was by Mr. H. Atkinson on the "Estimation of Potassium in presence of Sodium, Magnesium, Sulphates and Phosphates." The method described is based upon the relatively low solubility of potassium perchlorate in methyl alcohol compared with the solubilities of the perchlorates, sulphates, and phosphates of magnesium and sodium.

#### THE CHEMICAL SOCIETY.

Four papers were read at the meeting held on March 5, Dr. M. O. Forster presiding. "Amylases of the Cereal Grains: Rye," was presented by Mr. Julian L. Baker and Mr. H. F. E. Hulton, who have found that when ungerminated rye diastase acts upon starch paste or soluble starch at 50° C., crystallisable maltose and  $\alpha$ -amylodextrin only are formed. Rye diastase, however, is superior to barley in its capacity to liquefy starch paste, and it also gives a somewhat higher yield of maltose. The diastase of germinated (malted) rye gives from 76–86 per cent. of readily crystallisable maltose and a reducing dextrin having  $[\alpha]_D^{20} = +184$  and a reducing power of 10 (calculated as maltose). No intermediate products degradable by the further action of diastase (malto-dextrins) were obtained as when barley malt diastase acts upon starch paste under similar conditions.

In a paper on the "Hydrolysis of Cotton Cellulose," Dr. G. W. Monier-Williams described how he had converted cotton cellulose almost quantitatively into crystalline dextrose by direct hydrolysis. Ten grams of cotton wool were dissolved in 50 c.c. of 72 per cent. sulphuric acid and the viscous solution allowed to stand for one week. It was then diluted to 5 litres with water and the solution boiled for 15 hrs. After removal of the sulphuric acid with barium carbonate, the filtrate was evaporated to dryness under reduced pressure, being kept neutral to methyl red during the evaporation by repeated addition of sulphuric acid in small quantities. The sugar was extracted by methyl alcohol from the dry residue, and the methyl alcohol solution, after decolorisation with animal charcoal, slowly evaporated in a current of dry air. The residue was crystalline and contained 94.6 per cent. of dextrose, 3.3 per cent. of moisture, and 1.5 per cent. of ash. Calculated on the dry, ash-free cellulose, the yield of glucose amounted to 90.6 per cent. of the theoretical quantity.

The two other papers presented were:—"The Formation and Stability of Spiro-compounds. Pt. IV. The formation of ketones derived from open-chain and cyclic glutaric acids by the thermal decomposition of their calcium salts": G. A. R.



Kon; and "The Conditions underlying the Formation of Unsaturated and Cyclic Compounds from Halogenated Open-chain Derivatives. Pt. II. Products derived from  $\alpha$ -halogenated adipic acids": C. K. Ingold.

#### SOCIETY OF GLASS TECHNOLOGY.

The fourth annual general meeting was held at University College, London, on April 20, 1921. The report stated that the total members on the roll at the end of 1920 was 620, 128 members having been elected during the year. There was a small deficit on the year's working, due to the increased cost of printing the Society's Journal. After the election of officers, the new president, Dr. M. W. Travers, delivered his presidential address on "The Importance of Quantitative Investigation in dealing with Technical Problems in the Glass Industry."

Lord Moulton, who was to have presided at the dinner that evening, had insisted upon the observance of the principle that industrial processes must be based upon quantitative investigation, with the result that whenever his Department took over a factory it was almost invariably found possible to increase the efficiency of the processes carried out in it. Efficiency resulted from the economic use of money or credit, labour, materials, and energy, and was only possible when the losses under each heading could be accurately ascertained. The energy balance-sheet was really as important as the financial balance-sheet, and each should be drawn out with equal care. Just as it was impossible to check waste of money if one merely kept account of gross receipts and expenditure, so it was equally impossible to check waste of energy if one knew only the total fuel consumption in a furnace. Each furnace should be studied with a view to ascertaining whether unnecessary losses could be checked, the fuel consumption reduced, and the efficiency increased. The investigation of the nature of gas-producer tar, its composition, heat of formation, and its influence on the efficiency of heat exchange in the furnace should be investigated; also the actual temperatures of the gases as they entered and left furnaces. Furnace design should be based upon quantitative knowledge of the laws of the flow of gases through channels and spaces. The outlet flues from a pot furnace were not made small in order to keep the heat in the furnace, but because the velocity of the gas through a flue of small section was necessarily high, and when the velocity of the outflow was high, comparatively large fluctuations in the pressure in the furnace induced relatively small changes in the rate of outflow.

A paper on "Automatic Glass-feeding Devices," by Messrs. G. Dowse and E. Meigh, was read by the latter.

The third annual dinner of the Society was held on April 20 at the Hotel Cecil. Dr. M. W. Travers presided, and the guests included Mr. A. Chaston Chapman, Prof. F. J. Cheshire, Mr. Dennis Herbert, M.P., Mr. W. Ide, Master of the Glass Sellers' Company, and Mr. R. S. Whipple, president of the Optical Society. Replying to the toast of "The Society of Glass Technology," proposed by Mr. A. Chaston Chapman, Dr. Travers referred to the proposed legislation for the protection of the scientific branch of the industry. The view that the tariffs suggested were unsuitable and inadequate was held not only by the manufacturers, but also by the scientific and industrial worlds dependent upon supplies of chemical glassware. The only assistance which would enable this branch of the industry to continue was the total prohibition of all chemical glassware except under licence. It was a matter of great regret that electric-lamp bulbs had not been provided for in the Bill.

## NEWS AND NOTES.

### SOUTH AFRICA.

**The Fertiliser Industry.**—According to a statement made by Major C. C. Frye, of the Cape Explosives Works, Ltd., his company is about to extend greatly the manufacture of fertilisers and hopes within a few months to supply the whole of the fertiliser requirements of the Union, which are said to be approximately 100,000 tons per annum. The cost of the buildings and plant (which are now in course of erection) will not fall far short of £250,000. Every kind of fertiliser is to be sold by the company. —(*S. Afr. J. Ind.*, Mar., 1921.)

### AUSTRALIA.

**Starch Production from the Zamia Palm in New South Wales.**—As a result of investigations carried out by the Institute of Science and Industry, the Austral Starch Co. has been formed to produce starch from the Zamia palm. A factory has been erected on the south coast of New South Wales, and is about to commence operations. The company has been granted a licence to obtain Zamia bulbs from an area of 31,000 acres in the vicinity of Currawan, on condition that not less than 50 tons of dry starch shall be produced half-yearly. —(*Ind. Austral.*, Feb. 24, 1921.)

**Conservation of Wheat in South Australia.**—During the war the conservation of the South Australian wheat crop was placed under the control of a scientific committee of which Dr. W. A. Hargreaves, Director of the Department of Chemistry, S.A., was chairman. Two methods were used successfully:—(a) Treatment with Gas. The stacked grain was enclosed in sheds covered with "malthoid" made as airtight as possible. Then air, freed from oxygen by being passed through a furnace similar to a gas producer but designed to give carbon dioxide, was blown into the shed for three or four weeks to asphyxiate the insects. As many as 200,000 bags of grain have been treated at one time with success. (b) Heat Treatment. Dr. Hargreaves has modified the Poole and Steele machine, in which the wheat is passed through a hot zone at 140°–150° F.; this method was put into practice in 1918, and large quantities of wheat have been successfully treated by it.

### CANADA.

**Mining in British Columbia.**—On account of the continued low price of copper, the Granby Consolidated Mining, Smelting and Power Co. has reduced the wages of its employees, but the output of over 116 tons a month is still maintained. The Consolidated Mining and Smelting Co. is maintaining its zinc output with ore from the Sullivan mine, and its output of gold and copper has been increased. Now that the British American Nickel Corporation has closed its plant at Nickelton and the refinery at Deschenes, Que., the copper refinery of the Consolidated Co. is the only one at present operating in Canada. The concentrating plant of the Britannia Mining and Smelting Co., which was closed on November 30, 1920, owing to the low price of copper, has recently been destroyed by fire. With a daily capacity of 2500 t. of ore, the plant crushed 650,900 t. of ore during the first eleven months of 1920, and produced concentrates that yielded 8036 t. of copper. Practically none of the lead mines in the Slocan camp, the chief silver-lead mining area in British Columbia, is producing, but development work is proceeding at the Silversmith and a few other mines. A large belt of zinc-lead sulphides, 40 ft. wide and 3500 ft. long, has been uncovered at Bellevue, on the Illian River, Alice Arm district, where some of the small veins contain up to 150 oz. of silver and \$2.20 in gold per ton.

The iron and coal deposits of the Copper River district have been examined on behalf of British interests, and if the properties are acquired blast furnaces will be erected near Port Edward, and the Provincial Government will pay bounties of \$3 per ton of iron produced from domestic ores, and \$1.50 per ton of iron produced from foreign ores.—(*Canad. Chem. & Met.*, Apr., 1921.)

#### JAPAN.

**The Dextrin Industry.**—The manufacture of dextrin in Japan has been organised on a commercial scale only within the last three or four years. At present the cost of production is about \$5.53 per 100 lb., whereas the selling price on the Osaka and Kobe markets is roughly \$5.13 per 100 lb. for white and \$5.58 for yellow dextrin. Thus the manufacturers are not making a profit, and unless the cost of production is reduced by lowering wages and prices of raw materials the industry may not survive the present depression. There are three small factories situated in Osaka, Tokyo, and Kyushu, respectively, and the total output of the whole country is about 300 tons a month. Dextrin is mainly used in the textile industry, and the Japanese product is exported chiefly to Great Britain.—(*U.S. Com. Rep.*, Mar. 15, 1921.)

**The Dyestuffs Industry.**—The decline in prices subsequent to the armistice brought about severe depression in the Japanese dye industry, but imports of German dyestuffs were limited in amount, and some manufacturers resumed work late in 1919, the output in Tokyo-fu for the first five months of 1920 reaching a value of 1,503,000 yen (£153,306). The economic crisis which set in in April again curtailed production. Dyestuffs manufacture in Tokyo-fu began with Kryogene Black and was gradually extended to include Kryogene Brown, basic, acid, direct, mordant, and other dyes. The production of Kryogene and other dyes is given below:—

	Kryogene dyes.		Other dyes.	
	Long tons.	£	Long tons.	£
1918 .. ..	176.5	38,913	390.4	359,329
1919 .. ..	226.8	13,933	140.6	169,199
1920 (to May) ..	439.7	53,917	72.6	99,474

It will be seen that there was a considerable improvement in the output of Kryogene dyes up to the end of May, 1920, and that the production of other dyes was greater than in 1919, although much below that of 1918.—(*Bd. of Trade J.*, Apr. 21, 1921.)

#### BRITISH INDIA.

**Mineral Production in 1919.**—In his report for 1919, the Chief Inspector of Mines states that the output of coal was 21,759,727 tons, or 9.64 per cent. more than in 1918; nearly 70 per cent. of the production came from the Province of Bihar and Orissa. The coal used for coking amounted to 1,025,168 t., and 278,473 t. of hard and 308,565 t. of soft coke were made; the increased production of hard coke was chiefly due to the exceptional prosperity of the iron and steel industries. The output of coal per person employed was (a) below ground, 185 t., and (b) above and below ground, 114 t., compared with the average figures of 178 and 113, respectively, during the five preceding years. Owing to the cessation of the war demand, the accumulation of stocks, and the rise in exchange (factors which affected most minerals), the output of mica declined by 19.18 per cent. to 2084 t.; production was cut down and Government control of the mica industry terminated. Demand for manganese was on the whole worse than in 1918, and prices declined; the output was 1.16 per cent. higher at 420,184 t. The decline in the output of wolfram continued, and the output was 2905.4 t., a decrease of 19.5 per cent.; production remained normal during the first four months, but in May the purchase price dropped

from 72 to 30s. per unit, and by the end of the year the number of producing mines had fallen from 84 to 39. The output of copper ore was 32,756 t., compared with 3619 t. in 1918; 980 t. of copper was produced by the Cape Copper Co., which completed its mill and smelter at the Rakha mines in the Singhbhum district. There was a decrease of 43.81 per cent. in the output of gold (11,191 oz.), and a decrease of 3.37 per cent. in that of gems (158,577 carats), despite the demand at higher prices for rubies and sapphires. There were increases in the production of limestone, salt, slate, magnesite, hauxite, clay, tin, fullers' earth, samarskite, graphite, ochre, and molybdenite, and decreases in silver, iron ore, chromite, galena, steatite, harytes, apatite, and potash.—(*Ind. Tr. J.*, Mar. 18, 1921.)

#### FRANCE.

**Industrial Notes.—Metallurgy.**—Despite the uncertainty regarding the final financial settlement with Germany, the demand for pig iron and rolled products appears to indicate that trade in metallurgical products is reviving, and the keenness of demand for several finished products is a sure sign that consumers' stocks are running short. The question of exportation is again becoming prominent and a scheme is being considered involving an understanding between various iron and steel companies, and reduced transport charges to encourage exportation. It is also proposed to sell coal and coke from the Saar and Rhineland districts at special prices to exporting firms. The "Basset" process for the production of iron and steel direct from the ore (*cf. J.*, 1920, 304 x) is still attracting attention, but no definite statement can yet be made as the process is apparently still in the experimental stage. It is stated, however, that the cost of production is very low, so low in fact that were the cost to hold good in large-scale practice the process would revolutionise metallurgical production. Preliminary experiments are being carried out with 25-ton furnaces at Dennemont, near Mantes, and steel for rails has been produced. Three works with four furnaces, each of a capacity of 250 metric tons a day, are being erected in Normandy for a new company, "La Société des Aciéries Basset," which is working the process and whose activities may later be extended to Eastern France and even abroad.

An "Aluminium Week," to be held in Paris from May 21 to 29, is announced by the Société d'Encouragement pour l'Industrie Nationale. There will be an exhibition and a series of lectures to encourage the use of aluminium, magnesium, calcium, and sodium, and to promote new applications.

**Liquid Fuel.**—Pending a definitive arrangement, import licences will be required for liquid fuels as from May 1, 1921, and the following conditions will have to be complied with:—(a) The importer must purchase from the State a quantity equal to the amount imported; (b) a tax of 15 fr. per hectolitre will be levied to compensate the State for losses due to the depreciated value of its stocks; (c) 25 per cent. of the quantity of liquid fuel imported must be kept continuously in stock; and (d) the consumer must benefit from the lower prices accepted by the State.

**Chemical Industry.**—As yet there is no sign of improvement in the French chemical market, and several foreign markets, particularly Greece and South America, have been closed to France by German competition. Many German chemical products, particularly pharmaceutical chemicals, are even now finding their way into France, but a higher protective tariff and a general *ad valorem* duty on German imports should remedy this state of affairs. It is felt, however, that the real solution

lies in developing the French chemical industry so that it may turn out first-class products at minimum cost. The Cie. Cellulose Française d'Alfa, formed to manufacture paper and pulp from Algerian alfa, is contemplating the purchase of the Government's explosives factory at Toulouse, which has been practically dormant since the armistice. It would take 3-4 months to adapt the machinery to the company's requirements. The raw material would be shipped from Algeria to Cette, brought by canal to Toulouse, where it would be stored, and then carried by rail to the factory as required. The initial production is estimated at 20 metric tons of pulp from 40 t. of alfa, and the treatment would require 30 t. of coal, 1.5 t. of hydrochloric acid, and 3% of caustic soda; plant is available locally for the production of chlorine and caustic soda. It is estimated that paper pulp could be produced to sell at about one-half of the current price.

**Alsatian Potash Mines.**—The proposal to entrust the development of the Alsatian potash mines to a single company has been rejected by the Government Commission which is examining the question, and it has been decided to hand over the mines to four companies, which will have a common sales organisation both for home and foreign markets.

**Meeting of Civil Engineers in France.**—The summer meeting of the Société des Ingénieurs Civils de France will be held in France from June 18-24 next. The first five days will be devoted to a tour in Eastern France, including the battlefields of Verdun and the factories of Lorraine, and the last two days will be spent in Paris. Full particulars of the programme may be obtained on application to the offices of the British Section at 45, Great Marlborough Street, London, W. 1.

**The Perfume Industry in the Lyons District.**—Of late years the manufacture of artificial and natural perfumes in the neighbourhood of Lyons has expanded considerably, largely owing to the favourable geographical position and to the interest shown by the University of Lyons in offering a special course in perfumery. The Far East, Madagascar, and North Africa are the main sources of the raw materials used, but large supplies of lavender, iris, roses, etc., are obtained locally. The oil of verbena is transformed into violet, artificial iris and synthetic lily of the valley, oil of camphor into heliotrope, aniseed into anethol and artificial hawthorne, clove oil into vanilla, Guiana rosewood into bergamot, Madagascar faham into coumarin, etc. Lavender oil is produced in the Lyons district to the value of 10 million francs per annum, which, it is claimed, is 90 per cent. of the world's output. More than 12,350 acres are laid down to lavender in the district, but the cultivation of other flowers has been temporarily supplanted to some extent by that of food products. Synthetic perfumes are also manufactured and many of the factories which produced organic chemicals during the war now make perfumes. Thus, the toluene of a local firm is used for producing jasmine, artificial rose, as well as benzoate of soda, and the firm of Junillon, at Valence, makes a speciality of the production of synthetic musk from xylene. At present the industry is suffering from the trade depression, stocks have accumulated, and the principal buyers—America, England, and Japan—are not placing orders—(*U.S. Com. Rep.*, Feb. 28, 1921.)

#### UNITED STATES.

**The Beet-Sugar Crop in 1920-21.**—It is estimated that if weather conditions remain favourable the United States beet-sugar crop for 1920-21 will amount to 997,710 long tons, the largest crop ever produced in that country. Previous crops have been as follows:—1915-16, 780,550 t.; 1916-17, 732,700 t.; 1917-18, 683,000 t.; 1918-19, 674,900 t.;

and 1919-20, 653,000 t. Compared with last year, an additional 333,710 t. of home-grown sugar will probably be available, and as the United States is a large importer (*cf. J.*, 1920, 183 x), this decrease in requirements of foreign sugar may influence sugar prices in the world markets.—(*Ind. Tr. J.*, Apr. 1, 1921.)

**Gypsum in 1919.**—The quantity of gypsum mined in the United States in 1919 was 2,420,163 short tons, an increase of 18 per cent. over the output in 1918. All the producing States except Oklahoma showed an increase in output, ranging from 11 to 43 per cent., and large increases in value were also recorded. The distribution of the production was as follows:—Uncalcined: For Portland cement, 470,267 short tons; agricultural gypsum, 39,978 t. Calcined: Plaster of Paris, wall plaster, Keene's cement, etc., 1,393,141 t.; for glass manufacture, 14,677 t.; as boards, tiles, blocks, etc., 188,202 t. There were 56 active mines, quarries, and pits in the United States and Alaska, and these supplied 54 domestic calcining plants; most of the mills are equipped with kettles, but the number of rotary kilns is increasing, there being eight in 1919. Rock gypsum was mined in 41 localities, gypsite in 14, and selenite crystals in one. The imports of gypsum, mainly from Nova Scotia and New Brunswick, increased to 172,000 t. of crude and 10,000 t. of ground or calcined gypsum. Exports of plaster or wall board were valued at \$1,141,815, an increase of nearly 171 per cent. over 1918, England being the largest buyer (27 per cent.), followed by Japan, Canada, and Australia. The demand from South America continued to grow (*cf. J.*, 1920, 76 x).—(*U.S. Geol. Surv.*, Dec. 28, 1920.)

**Salt, Bromine, and Calcium Chloride in 1919.**—Owing to transport deficiencies and the cessation of war demand, the production and sale of salt in 1919 declined by 4.9 per cent. to 6,882,902 short tons. During the war output rose by 9.8 per cent. in 1915, 18.9 per cent. in 1916, and by 9.7 per cent. in 1917, the use of electrolytic chlorine in the manufacture of poison gases being mainly responsible for the increase. Fifteen States produced salt in 1919, the chief being Michigan, New York, and Ohio, and there were 102 operating plants in the whole country, the same number as in 1918. The production of rock salt was 1,642,057 short tons, mined by 21 firms in seven States, chiefly in New York and Kansas, and that of brine salt was 5,240,845 t., largely from Michigan and New York. Brine salt alone was produced in greater quantity than in 1918, and as it is usually sold to or produced by chemical works, the variations in its production may to some extent be regarded as an index of the state of the chemical industries. Imports of salt for curing meat and fish amounted to 62,821 short tons, and were obtained mainly from Spain, British West Indies, and England; in 1918 the import was 40,320 t., and the pre-war average 150,000 t. Exports totalled 119,416 t., the decrease of 13 per cent. being probably due to a return to normal conditions.

About 923 short tons of bromine, valued at \$1,234,969, was produced from the usual source, viz., bitters left after extracting salt from brine. Notwithstanding the cessation of the war demand, the production increased by 7.5 per cent. compared with 1918; the total given includes bromine marketed as bromides and other bromine salts.

The production of calcium chloride from natural brines was 26,123 short tons, a decrease of 1.9 per cent. Michigan, Ohio, West Virginia, and California remained the chief producing States of this type of calcium chloride (that produced in the ammonia-soda process being excluded); it contains from 2 to 6 per cent. of magnesium as an impurity (*cf. J.*, 1919, 28 x).—(*U.S. Geol. Surv.*, Mar. 26, 1921.)

## GENERAL.

**Increased Capitalisation of the German Chemical Industry.**—The reports of the largest German chemical companies, published last month, show that enormous increases of capital are contemplated. The capitals of the Farbenfabriken vorm. Friedrich Bayer and Co., of Leverkusen, the Farbwerke vorm. Meister Lucius and Bruning, of Höchst, and the Badische Anilin- und Sodafabrik, of Ludwigshafen, are each to be raised from 252 to 430 million marks, i.e., from about 12·2 to 21·5 million pounds at par (1 to 1·7 million pounds at the current rate of exchange); and the A.-G. für Anilinfabrikation ("Agfa") in Berlin proposes to raise its capital from 88 to 146 million mk. Thus, these four companies are to increase their combined capitalisation by 622 million marks (£31,100,000 at par). A large proportion of this sum is to be allocated to supply the future capital requirements of the German nitrogen works at Oppau and Merseburg, and the remainder is ear-marked for renewals and repairs, which in some works were not carried out during the war, and for re-converting certain factories to peace-time production. This policy of capital enlargement is also being pursued by other companies, e.g., the Fabrik Griesheim-Elektron proposes to increase its capital from 63 to 103 million mk., and Leopold Cassella and Co., in Frankfurt, from 113 to 135 millions. A comparison of these enormous figures, together with the sums expended on dividends in 1920-21 and the large amounts placed to reserves, with the corresponding figures for the period 1913-18, shows the undermining effect of the depreciated currency. The following are the official figures for the market values of German chemical securities in 1913, 1914, and 1918, in millions of marks:—

	1913.	1914.	1918.
Shares .. .. .	19·7	2·8	14·3
Debentures, etc. .. .	4·5	8·7	24

The division of the share capital and the dividend records of seven of the chief companies are given below:—

	Ord. shares.	Pref. shares.	Dividends (%)	1918.	1919.	1920.
	million mk.	million mk.				
Badische .. .. .	180	72	12	18	20	20
"Agfa" .. .. .	63	25	12	18	20	20
Elberfeld .. .. .	180	72	12	18	20	20
Griesheim-Elektron .. .	45	18	7	12	—	—
Höchst .. .. .	180	72	12	14	20	20
Kalle & Co. .. .. .	6	—	7	14	—	—
Weiler-ter-Meer .. .. .	16·0	6·7	10	12	15	15

In judging the significance of the large profits and dividend disbursements, it is necessary to take into consideration the depreciated currency and the fact that the plant and machinery were acquired before that depreciation took place. The shares of the "Agfa" company are quoted at 435-535 (for the 100 mk. share) so that the dividend represents only a very small return on the invested capital at its current value. Nevertheless, the figures show that the year 1920-21 was on the whole a satisfactory one. Recently the threatened "sanctions" have much increased the difficulties of production. For months past sales have been declining; in some works stocks have accumulated, and purchasers have been demanding larger and longer credits. Prices, too, have fallen without any corresponding decline in costs of production, and a few plants have temporarily closed down. The imperative necessity for making large allocations to plant depreciation has been strongly urged by Dr. Dnisberg, on the ground that the amounts now being written off are paper values, whereas the existing plant and apparatus were mostly purchased on a gold basis. For example, the depreciation of a plant which cost 1 million mk. five years ago would in normal times

be reckoned at 10 per cent. per annum; but this assumes that at the end of 10 years the plant could be renewed for another million. If, however, as at the present time, the cost of renewal is 10, 15, or 20 millions, it is obvious that a writing off at the rate of 100,000 mk. per annum is quite inadequate.

**"Aluminium and Bauxite."**—Like the other publications so far issued by the Imperial Mineral Resources Bureau, this pamphlet deals with the war period, in which are included the years 1913-1919. Its chief interest lies in the information it supplies as to the developments which have taken place in various new bauxite fields. Some of these had not reached a productive stage when war broke out and others had barely started to produce. At that time bauxite was only mined on a large scale in France and the United States, but British Guiana, Yugoslavia, and Rumsnia have since become important producers of the ore, whilst preparations for production are in an advanced state in Dutch Guiana and India. The pamphlet opens with some brief notes on the properties of aluminium and with a list of the chief aluminium minerals. The mineral cryolite is then discussed shortly, and here it is of interest to note that no deposits of this aluminium ore have been found other than the well-known bed in Ivigtut, in South Greenland. A general statement follows on the properties of various classes of bauxite, and attention is drawn to the qualities required according to the use to which the mineral is to be put, i.e., for the production of metallic aluminium, the manufacture of aluminium salts, of refractories or of abrasives.

The section which purports to deal with the uses of the metal will not convey much information, but from the first tables given the interesting conclusion may be drawn that the world's production of aluminium rose from 71,000 tons in 1913 to 170,000 tons in 1918. It is particularly noteworthy that the great increase in the demand which these figures indicate did not call into being any new successful process either for the production of refined alumina or for the reduction of alumina to metallic aluminium.

The occurrence and production of bauxite, the production, imports, and exports of aluminium, and other statistical matter are dealt with in a series of sections devoted to the following countries:—The United Kingdom, Gold Coast, Canada, British Guiana, India, Australia, France, Germany, Italy, Yugoslavia, Rumania, Spain, Switzerland, United States, and Dutch Guiana. Perhaps the most striking facts cited in this part of the pamphlet are the rapid development which has taken place in the productive capacity of the United States and the relatively still larger increase in the Norwegian smelting installations (Norway is not separately dealt with), whilst Germany has only become a serious producer of aluminium during the period under review. The discovery and exploration of extensive deposits of very high-grade ore in the Gold Coast are also notified.

The pamphlet concludes with a bibliography of the literature of the subject during the period dealt with. This is divided into sections on the Occurrence, Distribution, and Mining of Bauxite, The Metallurgy and Metallography of Aluminium, the Alloys of Aluminium, and, finally, the Uses of the Metal and its Compounds.

**The Bauxite Industry in British Guiana.**—The Demerara Bauxite Co., a British undertaking, has suspended operations on account of the small demand for the mineral in the United States, and an American company is also reported to have closed down for the same reason. The output of both companies has been shipped entirely to the United States, the quantity exported in January last being 8260 tons, valued at \$43,169.—(U.S. Com. Rep., Apr. 6, 1921.)

**Advance Statistics of Production, Exports and Imports of Certain Metals, etc. in 1920.**—The Imperial Mineral Resources Bureau has compiled the following advance statistics of production, exportation, and importation in respect of lead, zinc, silver, copper, tin, iron, and arsenic for the year 1920 (long tons throughout):—

**PRODUCTION IN 1920.***United Kingdom.*

	Long tons.
Pig iron .. .. .	8,007,900

*The Dominion of Canada.*

Copper .. .. .	36,230
Iron, pig, from Canadian ore .. .. .	67,740
Iron, ore, sold for export .. .. .	7,013
Lead .. .. .	15,172
Silver .. .. .	12,793,541 oz.
Zinc .. .. .	17,931
Arsenic, white, and in ore .. .. .	2,150

*Australia.*

Copper, bar .. .. .	24,069
In blister .. .. .	2,000
In ore exported .. .. .	115
Lead .. .. .	4,077
Lead bullion .. .. .	1,939
In lead concentrates exported .. .. .	4,122
Zinc .. .. .	9,655
Tin .. .. .	4,103
Iron, pig .. .. .	314,000
Arsenic .. .. .	1,202
Silver .. .. .	701,177 fine oz

*Federated Malay States.*

Tin .. .. .	34,934
-------------	--------

*Union of South Africa.*

Arsenic, white .. .. .	10
Copper ore and concentrates .. .. .	9,710
Iron ore .. .. .	2,280
Lead ore .. .. .	524
Tin .. .. .	2,130
Zinc ore and concentrates .. .. .	2,097
Silver .. .. .	892,203 oz.

*South Rhodesia.*

Arsenic .. .. .	390
Copper, blister .. .. .	2,775
Tin .. .. .	4
Silver .. .. .	158,982 oz.

*Northern Rhodesia.*

Copper (finished) .. .. .	130
Lead .. .. .	14,602
Silver .. .. .	5,601 oz.

*Nigeria.*

Tin concentrates .. .. .	6,022
--------------------------	-------

*Belgium.*

Raw steel (ingots, etc.) .. .. .	1,211,121
Finished steel .. .. .	1,055,530
Finished iron .. .. .	192,429
Zinc .. .. .	81,711

*France.*

Arsenic ore .. .. .	515
Copper ore .. .. .	367
Lead ore .. .. .	4,731
Zinc ore .. .. .	4,179
Tin ore .. .. .	20
Iron ore .. .. .	13,643,257

*Italy.*

Copper .. .. .	6,258
Lead ore (including argentiferous) .. .. .	35,741
Zinc ore .. .. .	96,514
Silver ore .. .. .	492
Cupreous iron pyrites .. .. .	4,329

*Cuba.*

Copper .. .. .	7,366
----------------	-------

*Mexico.*

Arsenic .. .. .	1,180
Silver .. .. .	33,112,571 fine oz

*United States.*

Copper— .. .. .	about 6,000
Smelter output from domestic ores .. .. .	551,339
Refinery production of new copper from domestic and foreign ores .. .. .	702,232
Iron ore .. .. .	87,773,000
Iron, pig .. .. .	36,750,000
Lead (refinery statistics) .. .. .	469,322
Silver .. .. .	56,564,504 fine oz.
Zinc (reports of ore-smelters only) .. .. .	423,368

*Chile.*

Copper .. .. .	106,000 to 110,000
----------------	--------------------

*Dutch East Indies.*

Tin .. .. .	13,994
-------------	--------

**EXPORTS IN 1920.***From the United Kingdom.*

To—	Pig iron.	Long tons.
Sweden .. .. .	.. .. .	19,320
Germany .. .. .	.. .. .	24,484
Netherlands .. .. .	.. .. .	31,172
Belgium .. .. .	.. .. .	181,191
France .. .. .	.. .. .	59,316
Italy .. .. .	.. .. .	80,652
Japan .. .. .	.. .. .	26,574
United States .. .. .	.. .. .	72,558
British East Indies .. .. .	.. .. .	14,241
Australia .. .. .	.. .. .	1,127
Canada .. .. .	.. .. .	9,689
Other countries .. .. .	.. .. .	63,131
Total .. .. .	.. .. .	579,455

*Lead.*

To—	Long tons.
Russia .. .. .	617
Sweden .. .. .	2,222
Germany .. .. .	421
France .. .. .	1,473
China (including Hongkong) .. .. .	780
Japan .. .. .	2,111
United States .. .. .	2,998
British South Africa .. .. .	247
British East Indies .. .. .	5,331
Canada .. .. .	7,144
Other countries .. .. .	11,233
Total .. .. .	34,623

*Zinc (crude and manufactures).*

Total .. .. .	10,308
---------------	--------

Copper (plates, rods, bars, blocks, cakes, etc., and manufactures of all sorts).

To—	Long tons.
Sweden .. .. .	252
Norway .. .. .	935
Germany .. .. .	40
Turkey .. .. .	878
Brazil .. .. .	464
Egypt (including Anglo-Egyptian Sudan) .. .. .	3,600
India .. .. .	7,990
Straits Settlements* .. .. .	305
Ceylon .. .. .	93
Australia .. .. .	1,261
New Zealand .. .. .	585
Netherlands .. .. .	533
Belgium .. .. .	1,409
France .. .. .	1,758
Italy .. .. .	4,101
British East Indies .. .. .	2,736
Other countries .. .. .	13,300
Total .. .. .	40,353

\* Including Federated Malay States and Labuan.

*Tin (blocks, ingots, bars and slabs).*

To—	Long tons.
Russia .. .. .	116
Sweden .. .. .	999
Germany .. .. .	214
France .. .. .	1,808
Turkey .. .. .	370
United States .. .. .	3,637
Canada .. .. .	1,113
Other countries .. .. .	5,073
Total .. .. .	13,335

*From the Dominion of Newfoundland.*

Iron ore .. .. .	510,600
------------------	---------

*From the Federated Malay States.*

Tin .. .. .	34,934
-------------	--------

*From the United States.*

Spelter from foreign ore .. .. .	28,500
Spelter from domestic ore .. .. .	86,000
Total .. .. .	114,500
Copper refined .. .. .	137,628
Ferro-manganese .. .. .	3,454
Ferro-silicon .. .. .	632
All other pig iron .. .. .	212,742

*From Bolivia.*

Tin (West Coast shipments only) .. .. .	29,542
---	--------

*From China.*

Tin .. .. .	11,795
-------------	--------

*From the Dutch East Indies.*

Tin shipments .. .. .	11,661
-----------------------	--------

*From Algeria.*

Iron ore .. .. .	1,096,527
Copper ore .. .. .	591
Lead ore .. .. .	12,598
Zinc ore .. .. .	12,598

**IMPORTS IN 1920.***Into the United Kingdom.**Pig iron (acid, basic, forge and foundry).*

From—	Long tons.
Sweden .. .. .	20,328
United States .. .. .	20,574
Other countries .. .. .	164,407
Total .. .. .	214,309

Zinc, crude and manufactures (including rolled sheets and plates).	
Total .. .. .	116,891

*Lead (pig and sheet).*

From—	
Spain .. .. .	58,149
United States .. .. .	9,756
Mexico .. .. .	1,040
Australia .. .. .	57,987
Other countries .. .. .	35,909
Total .. .. .	162,850

*Copper (bars, blocks, ingots, plates, sheets, etc.).*

From—	
Australia .. .. .	22,877
United States .. .. .	37,817
Chile .. .. .	13,323
Other countries .. .. .	38,461
Total .. .. .	112,478

*Tin (blocks, ingots, bars and slabs).*

From—	
Straits Settlements and Federated Malay States .. .. .	15,435
Australia .. .. .	1,704
Other countries .. .. .	11,613
Total .. .. .	28,742

*Into the United States.*

Totals—	
Zinc ore .. .. .	23,000
Spelter .. .. .	207
Tin .. .. .	50,563
Ferro-manganese .. .. .	59,254
Ferro-silicon .. .. .	13,900
All other pig iron .. .. .	123,201

**State Assistance for the Brazilian Vegetable-Oil Industry.**—The Brazilian Minister for Agriculture, Industry and Commerce has been authorised by the Government to negotiate with five leading firms for the erection of cottonseed presses and by-product plant in the States of Pernambuco, Parahyba, Ceara, Maranhao, and Pianhy. The Government will advance up to 75 per cent. of the outlay involved, provided that the plant is delivered ready for erection by December 31. The capacity of the cottonseed-oil plant must be at least 120 metric tons per month for small and from 430–600 t. for large works, and provision must be made for the eventual manufacture of soap, etc. Drugs, chemicals, plant, etc. required for the factories will, when imported, be given the same preferential treatment as goods imported for other State-supported enterprises. The five companies are as follows:—Sociedade Algodoeira de Nordeste Brasileiro, Companhia Parahybana de Beneficiamento e Prensagem de Algodao, Companhia Pastoral Agricola e Industrial Pianhyense, Philomemo Gomes Filhos, and Germano Boettcher. A large part of the production will probably go to the South-American markets, but doubtless other world markets will feel the competition.—(*Chem. Ind.*, Mar. 28, 1921.)

**The Title of "Civil Engineer."**—The Institution of Civil Engineers recently attempted to protect the title "civil engineer" but without success, on account of the difficulty of including engineers of high professional standing who were not members of the Institution. Now the Institution is applying to the Privy Council for a supplementary charter to enable it to confer the title "chartered civil engineer" as a recognised mark of professional status.—(*Min. Mag.*, Apr., 1921.)

**PERSONALIA.**

M. Georges Claude has been awarded the gold medal of the Société d'Encouragement au Progrès.

The readership in electrochemistry in the University of Manchester has been filled by the appointment of Mr. A. G. Lohley.

The "Sir Robert Hadfield Prize, 1921," has been awarded by the Council of the Institution of Mechanical Engineers to Mr. H. Moore for his paper on "A Small Ball-Hardness Testing-Machine."

Mr. J. E. Sears, jr., has been appointed Deputy Warden of Standards in succession to Major P. A. MacMahon, who has retired. Mr. Sears will continue to hold the post of Superintendent of the Metrology Department at the National Physical Laboratory.

Dr. E. F. Nichols has been appointed president of the Massachusetts Institute of Technology. He was formerly professor of physics at Yale University and is now director of research to the National Electric Lamp Association, Cleveland, Ohio. Dr. H. Hibbert, assistant professor of chemistry in Yale University, has been appointed to an associate professorship in applied chemistry. Dr. A. E. Taylor, professor of physiological chemistry in the University of Pennsylvania, and Mr. J. S. Davis, assistant professor of economics at Harvard, have been appointed directors of the Food Research Institute (*cf. J.*, 1921, 129 n).

The German Chemical Society has presented to Prof. Dr. Carl Duisberg a silver medallion in recognition of his great services to the Society and to chemical science.

The following academic appointments, etc. are reported from Germany:—Dr. E. Fromm, extraordinary professor of chemistry in the University of Freiburg (Breisgau), to be professor in the medical faculty at Vienna; Dr. A. Wisner to the professorship of chemical technology in the German Technical "Hochschule" in Brünn; Prof. Dr. Binz of Frankfurt to be professor of chemistry in the Agricultural "Hochschule" in Berlin; and Dr. J. Meyer, professor of chemistry in the University of Breslau, to succeed Prof. W. Biltz as professor in the School of Mines (Bergakademie) at Clausthal. Dr. Hermann Ost has retired from the chair of chemical technology in the Technical "Hochschule" at Hannover.

Dr. Henri St. George, one of the best-known chemists and bacteriologists in Eastern Canada, died suddenly on April 1.

Dr. T. Lynton Briggs, who joined this Society in 1885, died on April 3 at Flushing, New York, aged 63 years. He emigrated to America in 1886 and worked in the research department of the General Chemical Co. His father was one of the first to manufacture coal-tar dyes in England.

The death is announced of Dr. J. I. D. Hinds, chemist to the Geological Survey of Tennessee, at the age of 73. Dr. Hinds assisted in founding the American Chemical Society, and was professor of chemistry in Cumberland University and the University of Nashville and Peabody College.

The death is reported of Prof. T. Miyake of the Agricultural College of the Imperial University, Tokyo.

The deaths of the following German chemists and chemical technologists have recently been announced:—Prof. E. Kirchner, a well-known authority on the technology of paper manufacture, on February 14, in his 74th year; Dr. Carl Dittenhofer, one of the leaders of the German explosives industry, on March 15, in his 72nd year; and Dr. Alex. Bauer, formerly professor of general chemistry in the Technical "Hochschule" in Vienna, aged 85 years.



## PARLIAMENTARY NEWS.

### HOUSE OF COMMONS.

#### *The Budget.*

The Budget for 1921-22, introduced by Mr. A. Chamberlain, contained no new pronouncement of importance to chemical manufacturers. As previously announced by him, the excess profits duty, which yielded £219,000,000 in the past financial year, is abolished, hut income-tax, corporation tax, duties on beer and spirits, and postal rates remain unchanged. The corporation profits tax gave a disappointing yield of £650,000, instead of the anticipated £3,000,000. No additional taxation is to be imposed. The external debt has been reduced by £117,000,000 to £1,161,560,000, and the only debt outside the United States and Canada is £826,000 in Sweden, which will be paid this summer. Total revenue and expenditure last year were £1,425,985,000 and £1,195,428,000, and for the current year they are estimated at £1,216,650,000 and £1,038,728,000, respectively.—(Apr. 25.)

#### *Postal Rates on Scientific Journals.*

Mr. Rawlinson asked the Postmaster-General whether, in view of the importance of scientific journals and the fact that they cost no more to carry than newspapers of equal weight, he would allow scientific journals published fortnightly or monthly to be registered and carried at the same rate as newspapers.

Mr. Pease replied that similar suggestions had often been considered and rejected. The newspaper rate was already unremunerative, and the proposed measure would considerably increase the existing loss. Such an extension could not be restricted to scientific journals and would have to include magazines and other periodicals.—(Apr. 26.)

#### *Gretna Factory.*

Replying to Major W. Murray, Sir L. Worthington-Evans said that the future of Gretna Factory depended upon the settlement by the Government of the general policy regarding the national factories. The subject, which raised issues of the highest importance, was under consideration by the Cabinet, and a statement would be made as soon as a decision had been reached.—(Apr. 29.)

#### *Government Laboratory (Salaries).*

Answering Major Hills, Mr. Young said that the initial scales for future entrants to the chemical assistants' grade in the Government Laboratory would be £160 10s.—£200 for men and £140 10s.—£180 for women; the latter scale would come into force when any new women chemical assistants were engaged.—(May 2.)

#### *Power Methylated Spirits.*

Mr. Young, in reply to Sir W. Pearce, said that the allowance of 3d. per gallon on spirits used in making industrial or power methylated spirits represented the cost of excise restrictions to the home distiller. There was no reason to think that this allowance was insufficient and it could not be amended.—(May 2.)

#### *Rubber Output (Federated Malay Straits).*

In a written answer, Mr. Wood informed Major Boyd-Carpenter that proposals for legislation to restrict the output of rubber in Malaya had been submitted, but it had been decided that such a policy would involve financial liabilities which the Federated Malay Straits could not afford and would necessitate undue Government interference. Conditions must therefore be allowed to right themselves.—(May 4.)

#### *Dangerous Drugs Act.*

In answer to Mr. Gilhert, Mr. Shortt stated that the Committee on the Regulations under the Dangerous Drugs Act had nearly completed its inquiry. The report was expected shortly, and would be published.—(May 4.)

## REPORTS.

INTRODUCTION TO THE REPORT OF THE BRITISH MISSION APPOINTED TO VISIT ENEMY CHEMICAL FACTORIES IN THE OCCUPIED ZONE ENGAGED IN THE PRODUCTION OF MUNITIONS OF WAR IN FEBRUARY, 1919. Pp. 10. H.M. Stationery Office. 1921. [Cmd. 2d.]

The first section of this paper gives a list of the factories visited by Brig.-Gen. Hartley's Mission, together with a few notes on the "Interessengemeinschaft." German methods of manufacturing explosives and poison gases form the subject matter of the second. Mr. W. Macnab and Mr. S. I. Levy found that the German methods of making explosives were very similar to those in use in this country, the chief differences arising from the shortage of materials due to the blockade, e.g., plant for making ammonia from nitric acid, the use of paper crêpe in preparing nitrocellulose, and the substitution of glycol for glycerin (which was apparently not very satisfactory). The strong-acid method was employed for manufacturing picric acid, and *m*-nitrotoluene was separated from the mixed mono-isomers by fractional distillation *in vacuo*, thus allowing of the production of relatively pure TNT without later purification. Apart from these two instances, it may be said that the German methods of explosives manufacture, as seen, were not superior, and in some cases were inferior, to those used in the large English national factories. Dr. H. Levinstein and Mr. F. H. Carr were impressed by the fact that the bulk of the plant for producing poison gas had been used before the war in the manufacture of dyestuffs or pharmaceutical products; and also by the way in which the various stages of manufacture had been allocated among the factories according to the suitability of the available plant. The third section summarises the output of initial products, explosives and gases in the factories visited (*cf.* J., 1919, 398 R); the fourth deals with the military importance of the German chemical industry; and in the final part the position, in February, 1919, of the British and German chemical industries is reviewed [*cf.* J., 1919, 285 R, 303 R (Allmand), 408 R, 427 R (J. Allan), 468 R (Carr); and 1920, 47 R (Evans, Walpole)].

REPORT ON THE COMMERCIAL SITUATION IN SIAM AT THE CLOSE OF 1920. By T. H. LYLE, H.M. Consul-General, Bangkok. Pp. 42. Department of Overseas Trade. London: H.M. Stationery Office. 1921. Price 1s. 3d.

The bad trading and financial conditions prevailing throughout the world have also affected Siam, and there is little new to chronicle since the issue of the last report (*cf.* J., 1920, 300 R). A Siamese company, the Siam Coal Mining Co., Ltd., has been formed to exploit a concession near Trang, where coal has been found, but some difficulty is expected as the coal will not bunker, being liable to ignite spontaneously after a short exposure. An old-established British company has recently acquired temporary control of one of the tannery undertakings, with option to purchase should the venture prove promising.

The export of teak during the 12 months ended March 31, 1920, was practically double that of the previous year, but rice was exported in smaller quantities owing to the partial failure of the crop. The exports amounted in value to £15,718,170 (£1=11·28 ticals), and included:—Rice and rice products, 441,039 tons (£10,911,586); teak, 70,202 t. (£1,189,801); hides, £331,472; stick lac, 1063 t. (£156,430); sapphires and rubies, rough, £234,312; sugar, red, 4272 t. (£155,780); and salt, 62,428 t. (£80,733). Imports were valued at £12,272,967, of which the United Kingdom furnished 17·1 per cent. and the British Empire 74 per cent. Importation of iron, steel and machinery from the United Kingdom is steadily increasing, but Japan is losing ground, except in regard to enamelled goods, of which the imports have doubled. The chief articles imported, together with the percentage furnished directly by Great Britain, were as follows:—Glass manufactures, £64,621 (10%); china and earthenware, £146,539 (2·4%); chemicals, £51,384 (31·8%); copper, zinc, iron, lead, tin and other metals, £86,242 (35·3%); coal and coke, 15,545 t.; oils, other than petroleum, £126,211; medicines, £188,085 (8·9%); paper, £252,316 (32·1%); matches, £209,366; leather and manufactures of, £50,222 (14·4%); soap, £45,799 (71·2%). Prospects of improving British trade with Siam appear to be good. The report contains very full statistical tables of imports.

## COMPANY NEWS.

### LEVER BROTHERS, LTD.

The twenty-seventh annual general meeting was addressed by the chairman, the Rt. Hon. Lord Leverhulme, on April 22 at Port Sunlight. After referring to the good services of Mr. John Gray as vice-chairman, from which position he has retired to become chairman of Messrs. Gossage, of Widnes, and Messrs. John Knight, of London, the chairman commented on the precipitous deflation of prices which had occurred and expressed his belief that prices had now sunk to zero. He then spoke of the company's new capital arrangements (*cf.* J., 1921, 95 n, 134 n) and financial position, stating that the Niger Company had been purchased during the past year for 8½ million pounds, Price's Candle Co. for 1½ millions, Gossages and Crossfields for 1 million, and John Knight, Ltd., for about half a million, making a total of 11½ million pounds. Negotiations were entered into with the African and Eastern Corporation, but in view of the entirely changed conditions in West Africa, the scheme agreed upon had been abandoned by mutual consent. All new development work had been stopped.

Trade at present was in a most unsatisfactory state; the home trade of the soap industry had probably suffered less than that of most other industries, but export business was extremely quiet. There was a general slump in West African produce, and the position of the Niger Co. had been made more difficult owing to the mistaken policy of the directors of that company during the half-year prior to the change of control. With a view to supporting their own market, the Niger directors had made purchases of native produce through brokers in the open market, and when the slump came heavy losses resulted; during this time Lever Bros., anticipating the coming fall, were selling forward, and after the fusion of the two companies had been effected it was discovered that their profits on sales at high prices were offset by the losses of the Niger Co. The property acquired from the latter company was extremely valuable, and if it

ever became necessary to sell it—which is not contemplated—there would be no difficulty in obtaining a foreign purchaser at a price which would entail no loss to Lever Bros. Prospects for 1921 were bright. Helped by the withdrawal of Government interference and the excess profits duty, there was a great revival in the home trade and the beginning of a revival in the overseas trade.

During the war Lever Bros. and its associated companies (now 158) supplied the Government with over 60,000 tons of glycerin at the controlled price of £59 10s. per ton, compared with £200—£500 per ton paid for the imported product, and it was estimated that the total advantage to the Government of having this large home supply represented a money gain of over £10,000,000. Although by virtue of the production of glycerin, the soap industry was a "key" industry, there was no desire to keep out overseas competition by protective tariffs; absolutely free-trade conditions were to be preferred. For the first time in its history the company had experienced a strike. The employees had recently agreed to a reduction in wages; the total sum disbursed in wages by the parent and associated companies in 1920 was £7,707,031, whereas that paid in dividends to the ordinary shareholders was £456,000. During the period July, 1914, to December, 1920, £3,760,814 had been paid in income tax. In reply to questions, Lord Leverhulme said that the recent issue of debentures was made to reduce the outstanding liabilities, which amounted to about £8,000,000, and that the company's claim for repayment of excess profits duty would be a very heavy one.

**ASSOCIATED PORTLAND CEMENT MANUFACTURERS, LTD.**—The accounts of this company for 1920 show a record revenue, after allowing for taxation, of £952,531, a sum which includes investments in associated companies, etc. Issued capital amounts to £4,422,040 and debentures stand at approximately £4,000,000. There is a general depreciation fund of £1,050,000 and other reserves amount to £661,000. The dividend for the year is 10 per cent. or 2 per cent. better than for the previous year and £221,561 is carried forward, against £143,51 brought in. The position and prospects of the cement industry were outlined by the Hon. F. C. Stanley at the annual meeting held in London on April 26 (*cf.* J., 1921, 156 n).

**THARVIS SULPHUR AND COPPER CO., LTD.**—Addressing the annual meeting of this company on April 26 in Glasgow, Mr. W. P. Rutherford, who has succeeded the late Lord Glenconner as chairman, attributed the very low prices ruling in the copper market to the existence of an unprecedented amount of scrap material and the general lack of demand. A gradual improvement, however, was to be expected, and the company's mining resources in Spain were ready to meet whatever demand arose for its ores when markets returned to their pre-war activity; but as long as the present stagnation lasted the company would restrict expenditure as far as possible. A dividend of 15 per cent. was passed.

**THE SALT UNION, LTD.**—The annual meeting of this company was held on April 19 at Liverpool. Mr. G. H. Cox, the chairman, in a review of the position and prospects of the salt industry, said that output had been very seriously reduced, the number of pans now working being only 45, compared with 245 a year ago. Thanks to better shipping facilities, the Eastern trade had improved in 1920, no less than 100,000 tons of salt having been delivered to Calcutta, besides 5000 t. to Rangoon. But prospects in the Indian market had darkened owing to the competition of German crushed salt.

from Hamburg and solar salts from Port Said, Spain, and the Red Sea. No English salt had been shipped for two months, but now a limited business was being done, and prices were more satisfactory. Exports to Japan amounted to 6000 t. in 1920, and recently 18,000 t. had been shipped, in the production of which the Weston Point vacuum plant had furnished 4593 t. of salt in one week. Prospects in Scandinavia and in many other export markets were adversely affected by German competition, which was much helped by the abnormal position of the German exchange. The good export trade in 1920 justified the company in maintaining the dividend of 15 per cent. on the ordinary shares.

## TRADE NOTES.

### BRITISH.

**South African Imports of Chemicals in 1919.**—The measure of South Africa's dependence upon other countries for supplies of chemicals and drugs may be gauged from the following table of imports during the year 1919. The third column gives the percentage weights in long tons (or volumes) of the substances obtained from the countries indicated:—

	Quantities.	Countries of origin Per cent.
acetone, methyl alcohol..	galls. 8,661 ..	U.K. 24.3, U.S. 30, Canada 45.
Acetic..	.. .. 1,745 ..	U.K. 34.8, U.S. 27.4, Japan 37.7.
Boric ..	tons 18.8 ..	—
Nitric ..	.. .. 1.7 ..	—
Phosphoric ..	.. .. 381 ..	U.S. 92, U.K. 7.7.
Sulphuric ..	.. .. 7.1 ..	—
Tannic ..	.. .. 1.9 ..	—
salts (unenumerated) ..	.. .. 247.3 ..	U.K. 100.
ammonia, anhydrous ..	.. .. 52.8 ..	U.S. 40.6, U.K. 30.7, Australia 28.6.
ammonium carbonate ..	.. .. 29.5 ..	—
ammonium nitrate ..	.. .. 0.6 ..	—
raz ..	.. .. 94.6 ..	—
aluminium carbide ..	.. .. 2,756.7 ..	Can. 84.2, U.S. 5.3, U.K. 2.5, Sweden 3.2, Norway 2.5.
lithia of lime ..	.. .. 213.5 ..	U.K. 97.2.
infectants ..	.. .. (386,610) ..	U.K. 93.6.
acidicides ..	.. .. (87,127) ..	U.K. 45.4, U.S. 27.5, Australia 25.3.
um arabic ..	tons 34.9 ..	—
tharzo ..	.. .. 26.7 ..	—
agnesium sulphate ..	.. .. 69.5 ..	—
official preparations ..	.. .. (1,096,247) ..	U.K. 63.2, U.S. 31.6.
ter drugs and chemicals ..	.. .. (1,099,318) ..	U.K. 67.8, U.S. 18.5.
ium ..	lb. 896 ..	Turk. 37.5, Ind. 35.7, Persia 23, U.S. 81.2.
ash compounds ..	tons 164.4 ..	—
assium cyanide ..	.. .. 2.2 ..	—
da ash ..	.. .. 1,439.9 ..	U.K. 79.5, Brit. E. Afr. 19.5.
da, caustic ..	.. .. 1,763.9 ..	U.K. 62.7.
dium arsenite ..	.. .. 426.9 ..	U.K. 50, Austral. 50.
da cpds. (unenum.) ..	.. .. 456.5 ..	U.K. 93.7, U.S. 7.2.
dium cyanide ..	.. .. 1,294.7 ..	U.K. 94.4, Germ. 5.6.
dium silicate ..	.. .. 11.6 ..	U.K. 98.

Items in italics are given in values (dollars) only.

By value the United Kingdom contributed about 1 per cent. of the total imports.—(U.S. Com. Rep., ar. 12, 1921.)

**Mauritius in 1919.**—The sugar industry was very prosperous in 1919, and prices exceeded all previous standards. The high-water mark of production has, however, been reached, as practically all the areas suitable for cane growing are already under the crop, and any increase in production will probably come through improved methods of cultivation and milling. Of the total area under cultivation, 8,091 acres, no less than 175,923 acres was under gear. Cultivation of aloes fibre declined, and the agency to plant sugar-cane on land formerly occupied by aloes continued. Increasing attention

was paid to the possibilities of growing sisal, and trial plantations were established, but the planters were not enthusiastic. Sugar-cane megass is an excellent paper-making material, but hitherto it has been used as fuel in the sugar factories, and as such its value is only equivalent to that of the fuel it replaces. The value of imports during 1919 was Rs. 47,037,024, of which the United Kingdom supplied 24.3 per cent., compared with 16.3 per cent. in 1918 (rupee=1s. 4d. at par). Imports of coal, machinery, sulphate of ammonia, and linseed oil from the United Kingdom declined, and the high freight rates drove some of the trade to British possessions, particularly South Africa. The value of imports from the United States and Japan also diminished. Exports were valued at Rs. 127,806,321, of which 75 per cent. went to the United Kingdom; they included:—Sugar, 302,826 metric tons; aloes fibre, 2176 t.; copra, 234 t.; coconut oil, 2411 hectolitres; vanilla, 2995 kg.; and molasses, 103 t. (cf. J., 1920, 437 R).—(Col. Rep.-Ann., Cmd. 1103-5, Feb., 1921.)

### FOREIGN.

**Foreign Trade of Japan in 1920.**—A comparison of the Japanese trade statistics for 1919 (cf. J., 1920, 327 R) and 1920 shows that the value of the total imports rose from £221,692,930 to £238,286,178, and that the total exports declined from £214,084,842 to £198,735,678, the decrease being accounted for mainly by semi-manufactured goods. The values of imported and exported chemical products are given below, together with the percentage increases or decreases over those in 1919, the yen being taken at 2.01s.:—

	Imports, 1920.	Inc. or dec. over 1919.
	£	Per cent.
Sugar ..	6,142,440 ..	+ 3.5
Salt ..	1,673,922 ..	- 13.7
Oil-bearing materials ..	1,601,262 ..	- 21.9
Hides and skins ..	1,975,542 ..	+ 25.3
Rubber and gutta-percha, crude ..	1,368,234 ..	- 22.7
Chile saltpetre ..	2,523,480 ..	+ 78.8
Ammonium sulphate, crude ..	2,029,944 ..	- 27.6
Oilcake ..	15,392,718 ..	+ 11.6
Coal ..	2,031,126 ..	+ 7.1
Ores ..	1,627,614 ..	- 23.7
Leather ..	857,208 ..	+ 53.3
Paraffin wax ..	770,202 ..	+ 9.1
Caustic soda and soda ash ..	1,185,444 ..	- 20.0
Coal-tar dyes ..	1,374,778 ..	+ 44.0
Paper-pulp ..	1,344,870 ..	+ 23.4
Iron, pig, rod, tube, plate, etc., ..	26,342,112 ..	+ 12.4
Lead, ingot, slab ..	725,118 ..	- 34.8
Tin, ingot, slab ..	758,982 ..	- 7.0
Petroleum ..	2,169,540 ..	- 1.9
Paper ..	1,794,282 ..	- 4.6
	Exports, 1920.	Inc. or dec. over 1919.
	£	Per cent.
Starches ..	509,694 ..	- 60.8
Sugar, refined ..	3,121,592 ..	+ 41.5
Coal ..	4,610,100 ..	+ 19.9
Oils, vegetable ..	1,092,628 ..	- 18.9
Camphor ..	506,430 ..	- 27.0
Iron ..	1,503,684 ..	- 30.6
Copper ..	1,294,278 ..	- 51.5
Leather manufactures ..	319,566 ..	- 52.5
Soaps ..	465,426 ..	+ 4.3
Matches ..	2,902,206 ..	- 13.7
Papers ..	2,359,056 ..	- 8.9
Cement ..	1,025,618 ..	+ 53.7
Pottery ware ..	3,208,512 ..	+ 39.0
Glass, and manufactures of ..	2,350,873 ..	+ 18.0
Rubber tyres ..	617,734 ..	+ 12.7

The importation of precious metals increased, in spite of the fact that the unfavourable balance of trade in 1920 was five times as great as in 1919.—(U.S. Com. Rep., Feb. 16, 1921.)

**Austrian Foreign Trade in 1920.**—The foreign trade of Austria for the fiscal year 1920 amounted by weight to 5,479,230 metric tons, of which 1,992,990 t. represented the trade between Austria and Germany. Germany supplied 40 per cent. of the total imports (4,502,170 t.) and took 20 per cent. of the exports (977,060 t.). Certain of the principal im-

ports and exports are given below, in metric tons, for the period July 1, 1919, to June 30, 1920:—

	Imports.	Exports
	Metric tons.	
Coal .. .. .	2,191,200	—
Celluloid .. .. .	110	—
Magnesia .. .. .	—	55,200
Graphite .. .. .	—	6,880
Glass and glassware .. .. .	18,500	—
Cement .. .. .	—	10,600
Pottery .. .. .	27,700	21,200
Chemical materials and products .. .. .	43,600	35,320
Dyes, varnishes, and medicines .. .. .	2,100	—

The total values of the imports from, and exports to, Germany were 13,427 million and 5,419 million kronen, respectively (krone=10d. at par, now about 0·18d.).—(*U.S. Com. Rep., Mar. 21, 1921.*)

**Swiss Imports of Chemicals and Oils in 1920.**—Importations of chemicals and oils into Switzerland in 1913 and 1920 were as follows:—

	1913.	1920.
	Metric tons.	
Sulphur, lump, etc. .. .. .	33·88	38·62
Turpentine oil .. .. .	19·82	17·05
Caustic soda and potash .. .. .	89·06	115·71
Sulphuric acid .. .. .	96·11	132·8
Copper sulphate .. .. .	21·03	8·68
Benzine .. .. .	160·88	349·2
Aniline .. .. .	12·02	23·46
Aniline compounds .. .. .	8·87	15·23
Alcohol, denatured .. .. .	13·36	15·91
Linseed oil .. .. .	47·69	20·9
Olive oil, olein .. .. .	6·27	5·54
Fats and oils, liquid, crude .. .. .	28·67	18·26
Coconut and palm oil, crude .. .. .	21·91	246·12
Petroleum .. .. .	629·42	122·03
Lubricating oil .. .. .	138·9	187·17
Bone meal, raw phosphates .. .. .	188·85	651·45
Basic slag .. .. .	557·93	166·31
Potash fertilisers .. .. .	132·41	167·32
Other fertilisers .. .. .	263·09	—

—(*Chem. Ind., Mar. 28, 1921.*)

## GOVERNMENT ORDERS AND NOTICES.

**PROHIBITED EXPORTS.**—The following have been removed from the list of prohibited exports, as from April 28:—(1) Potash, caustic, and articles containing caustic potash; (2) Potassium carbonate and mixtures containing potassium carbonate.

**ORDERS UNDER THE GERMAN REPARATION (RECOVERY) ACT, 1921.**—Order No. 6, dated April 21, excludes from the provisions of the Act articles produced or manufactured in the Saar Basin.

By Order No. 7 of April 28, any article proved to have been delivered by Germany to any Allied or Associated Power by way of reparation is exempted from the provisions of the Act.

Order No. 8, of May 3, exempts any article imported before June 15, 1921, provided it is essential for completing and working machinery or plant partly delivered before March 8, 1921, or is an essential part of German machinery installed in the U.K. before that date; and if patterns, drawings, or designs were sent to Germany before March 8, provided that their value is not less than 20 per cent. of that of all articles to be manufactured under the contract.

Order No. 9, dated May 3, exempts any article sent from the U.K. to Germany, or returned unaltered to the U.K. from Germany, provided that the consignor in the U.K. still retains the ownership.

In Order No. 10, of May 3, sections (a) and (b) of Order 1 (*cf. J., 1921, 155 B*) are recapitulated, and the date up to which importation is allowed is extended to June 15.

**WOMEN AND YOUNG PERSONS (EMPLOYMENT IN LEAD PROCESSES) ACT, 1920.**—Draft Orders have been issued by the Home Secretary under Section 2 of the above Act, providing that any women or

young persons employed in any process in which dust or fume from a lead compound is produced or in which such persons are liable to be splashed with any lead compound must be examined once in every three months by the district Certifying Surgeon appointed under the Factory and Workshop Act, 1901, and that suitable cloakroom, mess-room, and washing accommodation shall be provided. These proposed Orders are based on the requirements in existing Regulations and Orders for other processes.

**EMERGENCY REGULATIONS.**—By Royal Proclamation of April 30, the state of emergency proclaimed on March 31 and the Emergency Regulations of April 1 were continued.

The supply of coal to breweries and distilleries, except to certain yeast-producing distilleries, has been prohibited.

## OFFICIAL TRADE INTELLIGENCE.

(From the Board of Trade Journal for April 28 and May 5.)

### OPENINGS FOR BRITISH TRADE

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W. 1, from firms, agents or individuals who desire to represent U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number.

Locality of firm or agent.	Materials.	Reference number.
Australia .. .. .	Skins, hides, glove leather .. .. .	412 20/7-543
British West Indies .. .. .	Cement, paper .. .. .	527
China .. .. .	Chemicals .. .. .	567
Canada .. .. .	Gelatin .. .. .	529
" .. .. .	Soap, disinfectants, china .. .. .	521
" .. .. .	Crockery, china .. .. .	525
" .. .. .	Floor oil, washing compounds .. .. .	504
" .. .. .	Polishes .. .. .	563
South Africa .. .. .	Tinplate .. .. .	566
Belgium .. .. .	Window glass, steel plates .. .. .	529
" .. .. .	Paper .. .. .	—
" .. .. .	Pig iron, minerals, chemicals, cement, glass, china, crockery, paint, varnish, paper, leather .. .. .	511
Danzig .. .. .	Edible oils and fats, industrial and mineral oils .. .. .	5294
France .. .. .	Copra, groundnuts, oilseeds, crude drugs, hides, skins, tanning materials .. .. .	532
Greece .. .. .	Mexican fibre .. .. .	533
Italy .. .. .	Crude linseed oil .. .. .	534
Netherlands .. .. .	Fertilisers .. .. .	535
Norway .. .. .	Chemicals .. .. .	537
" .. .. .	Tinplate, steel sheets .. .. .	538
" .. .. .	Olive oil, animal fats, sugar, syrup, soap .. .. .	539
" .. .. .	Hides .. .. .	540
" .. .. .	Technical and edible oils .. .. .	541
Spain .. .. .	Chemicals, china, glassware .. .. .	542
" .. .. .	Chemicals, pharmaceutical products, colours, paint, varnish, metals, fine earthenware .. .. .	546
United States .. .. .	Earthenware, china .. .. .	548
Argentina .. .. .	High-grade varnish and enamel .. .. .	549
Cuba .. .. .	Earthenware .. .. .	550
" .. .. .	Plate and window glass, tinfoil, crown corks .. .. .	552
Mexico .. .. .	Paint, varnish .. .. .	562
" .. .. .	China .. .. .	593

\* The Official Secretary, Commercial Information Bureau, Australia House, Strand, London, W.C. 2.

† The Canadian Government Trade Commissioner, 73, Nassington Street, London, E.C. 2.

**MARKETS SOUGHT.**—A Canadian tannery able to export leather for bookbinders and bag makers, wishes to hear from importers in the U.K.

A Canadian tannery able to export 1000 sides per week of chrome leather in black and colours desires to get into touch with U.K. importers.

A firm at Montreal able to export asbestos wishes to hear from importers in the U.K.

[Inquiries to the Canadian Government Trade Commissioner.]

#### TARIFF. CUSTOMS. EXCISE.

**Australia.**—Drugs and chemicals, not elsewhere included, when packed for use in the household are dutiable under Item No. 79, and medicines for similar use under Item No. 285.

**Austria.**—As from April 15, customs duties when paid in bank notes must be paid at 80 times the rates specified in the tariff.

**Belgium.**—Sulphuric ether may be imported without licence as from May 2.

**British Honduras.**—The import duty on fuel oil has been doubled, and the export duty on alcoholic beverages reduced.

**British India.**—The valuations of raw hides and skins for export duty are set out in the issue for May 5.

**Cyprus.**—The export is prohibited to all destinations of, *inter alia*, chemicals, drugs, oils, copper, silver and gold.

**Czechoslovakia.**—A copy of the export free list may be seen at the Department, 18, Queen Anne's Gate, S.W. 1.

**Denmark.**—A "dispensation duty" is charged in connexion with the issue of licences for the import of goods containing sugar. A complete schedule of such duties may be seen at the Department.

**Ecuador.**—The export duty on ivory nuts is fixed, as from January 1, at 2 centavos per kg. for unshelled and 3 centavos per kg. for shelled nuts. Certain additional export duties are also levied thereon.

**Egypt.**—Exports to Russia are no longer subject to licence except in respect of goods the export of which is prohibited.

Recent import tariff valuations affect, *inter alia*, copper, brass, tin, lead, zinc, phosphor bronze, antimony, and quicksilver.

**France.**—The export duty on dephosphorisation slag has been withdrawn as from April 3.

**Germany.**—Import licences are no longer required for animal manures, and many seeds and condiments.

Export prohibitions have been withdrawn from raw cocoa, lemon juice, certain spices, white of egg, cocoa butter, rice starch, vinegar, certain flavouring essences and chemically prepared foods, etc.

**Do. (Occupied Territory).**—Not more than 5 kg. of goods (with some exceptions, including pharmaceutical products and synthetic dyes) may be imported by parcel post without licence.

**Iceland.**—The import is prohibited of chocolate, chinaware, liquorice, and perfumery. The import of almost all other goods is free.

**Italy.**—Until further notice all goods (with certain exceptions) may be imported by parcel post without licence.

**Luxembourg.**—All goods may now be exported without restriction with a few exceptions, among which are chemical and natural manures.

**Mexico.**—The export duty on sugar has been abolished.

**Netherlands East Indies.**—A copy of the new tariff law may be seen at the Department.

Increased import duties are payable on wares of iron, steel, tin, lead, copper, zinc, leather, and paper.

Modified export duties are payable on mineral oil and by-products thereof, and new duties on rubber, quinine, copra, and coconut oil.

**New Zealand.**—Export prohibitions have been withdrawn from glue, gelatin, glue pieces, hide pieces, and similar goods for the manufacture of glue and gelatin.

**Nigeria.**—The import duties on spirits have been increased.

**Norway.**—Export prohibitions have been withdrawn from certain hides and skins, leather, and pyrites.

**Paraguay.**—The import duties on starch, creolin, matches, common soap, and candles have been modified.

**Rumania.**—The export is prohibited of caustic soda, copper, brass, flax, flaxseed, grease, iron ore, pyrites, lard, animal fats, paper, paraffin wax, crude petroleum, petroleum residues, and zinc.

Special authorisation is required for the export of bones, cellulose, cement, ferromanganese, old iron, tarred paper, petroleum products not specified above, and shoemakers' glue and starch.

**South Africa.**—The import duties on beer and spirits have been increased.

**Spain.**—The export is permitted without restriction of all kinds of paper, cardboard and pasteboard.

The restrictions on the export of certain hides and skins have been relaxed.

The surcharge of customs duties when paid in Spanish silver or notes of the Bank of Spain is fixed for the month of May at 37.79 per cent.

**Sweden.**—Export prohibitions have been withdrawn from certain oilcakes and cattle foods.

A copy of the regulations affecting the testing of leather substitutes may be seen at the Department.

It is proposed to increase the customs duties on china, earthenware, glass, iron and metal goods.

**Tunis.**—Export prohibitions have been withdrawn from essence of turpentine and celluloid.

#### REVIEWS.

**FACTORY ADMINISTRATION AND COST ACCOUNTS.** By EDWARD T. ELBOURNE. *New edition.* Pp. xx. + 811. (London: Longmans, Green and Co. 1921.) Price 45s. net.

One of the most interesting developments of recent years has been the attempt to place factory costs and management on a scientific basis. Just as in the chemical industry there has been a constant progress in the replacement of "rule of thumb" and empirical methods of manufacture by those based on exact knowledge, so in other branches of industry has there been an endeavour to replace rough estimates by accurate costings and to base management more on known statistical figures than by observation—more or less cursory—of the work in hand. There has been, moreover, a marked contrast in the progress of various industries, for while the engineering and heavy chemical trades have been firmly established, the dyestuff and fine chemical industries are still in the embryonic stage. In engineering the systems of costing and of factory control have been developed to a state of great efficiency, but only recently has the value of chemical control of material begun to be recognised. In the chemical industry the converse is possibly true, for there, after years of propaganda, the value of research and laboratory control is well established and extensively practised, but the vital necessity of accurate costings and the value of scientific management is but just beginning to be realised. Although the principles of control, chemical or otherwise, of experiment and research, of costing and of management are the same, whatever may be the industry, there is

a great difference in their application. Thus, in engineering, labour is the all-important item, and material costs are small in comparison, as, for instance, take the cost of a £1000 motor car and contrast the value of the metal used and of the labour involved: whereas in the chemical industry the value of the material employed is often many times that of the labour. This results in a different outlook in the two industries. In engineering, labour costs and rate of production are the factors most eagerly scrutinised, whilst in chemistry yields play the most important part in factory cost. Again in regard to expenses, whilst many are common to both industries, in engineering the cost of machinery and its output may be contrasted with the laboratory and supervision expenses in the chemical industry. It follows that though both the engineers and the chemists are agreed on the principles of costing and management, the application of these will be different in each case, and not only will they differ in each industry, but in each particular branch.

The chemical manager, therefore, who wishes for information on these important points can only expect to find general principles in the literature available, and must be prepared to work out the particular application suited to his works. He will be in much the same position as he finds himself with regard to his own science, for whilst he can get certain information from published chemical literature, he must amplify this and work out his methods in his research and technical laboratories and his own drawing office. With a recognition of these factors, many chemical managers welcomed the appearance of the first edition of "British Factory Administration and Accounts" in 1914, and will turn eagerly to the 1921 edition\* to see what help in their industry they may derive from it. The author of these books has had great experience in some of the largest engineering undertakings in the world, and therefore speaks with authority on the practice in this industry, but the chemical manager will turn to the book to select those systems which can be successfully applied in his own particular works.

The preface promises well, emphasis is laid on the fact that the book deals essentially with principles and that practical applications are given by way of illustration; this underlying idea of the work is fully in keeping with the remarks previously made. It is also clear that the book is intended as one for reference. In this review it is intended to consider it from the standpoint of its value to one in charge of a chemical works. It consists of over 700 pages, with an excellent index of 60 pages. These are divided equally (approximately) into Works Management and Works Accounting, and the subjects are dealt with most exhaustively from an engineering point of view. The greater portion of the work, unfortunately for the chemical manufacturer, is occupied with the illustrations rather than by a discussion of principles, and these illustrations will, it is feared, prove of very limited application in the management of chemical works. It is proposed, therefore, to deal with these principles rather than with details of forms and methods specially applicable to engineering; also to consider the subjects as they are divided in the book itself.

1. *General Administration.*—There is very little in this section which will be helpful to the manager of a chemical works, though here and there items of interest crop up; for example, on page 18 the question of buying material is entirely looked at from the engineering standpoint, whereas in a chemical works the quality of the article very often determines its price and certainly its value to the manufacturer. On page 64 there is a very interesting statement regarding the relation of works expenses to direct wages, which is worth the careful

attention of all concerned. The fallacy of using a ratio of works expenses to the direct wages bill as a measure of the efficiency of management is pointed out. It may be remarked that in a perfectly organised works where machinery is used to such an extent that the amount of direct labour is practically negligible, the proportion of expenses to direct labour would be enormous and, to the uninitiated, might excite the utmost alarm as to the efficiency of the management. There is a great deal of useful and general information in this section to those dealing with the administrative side of the business, such as sales, organisation of the office staff and legal points relating to the question of insurance, etc.

2. *Works Management.*—Divided into three sections of Production, Labour, and Material Control.

*Production.*—This section is a chapter of 12 pages relating to buildings and plant. It might very well have been extended, as it is a subject of very general interest, whereas the other chapters relate almost entirely to conditions existing in the engineering trade. The section on buildings and plant is so essentially a question in which every manufacturer is interested that it is a great pity it was not further elaborated.

*Labour Control.*—This relation of engineering employers with their employees and of the unions concerned has been developed to such an extent that the chemical trade will be very glad to obtain from them all the information possible, and therefore this section on labour administration will prove of very great interest to the chemical works manager, and will also prove most valuable for the purpose of reference. It is certainly one of the most useful sections in the book.

*Material Control.*—This section deals with the methods used in the engineering industry, and will prove but of very limited use to those engaged in the chemical industry.

3. *Works Accounting.*—Divided into two sections:—(a) Administration Records, (b) Cost Accounts, followed by specimen forms of works routine, with a complete index.

(a) *Administration Records.*—This is an extremely important section, and its relation to the all-absorbing question of cost accounts is well defined. In contradistinction to the earlier part of the work, this section is of general interest, and will well repay careful study by all concerned in factory management and accounting. It contains a full account of matters of importance to every factory, such as employment records and methods for the accurate keeping of all accounts relative to wages, stock and plant, as well as much valuable information as to stocktaking and depreciation and valuation of plant. Whilst the portion relating to depreciation is more particularly devoted to engineering, the whole section contains such a mass of general information that it will prove of the utmost service as a guide to what should be done in organising or reorganising a works, as well as a useful reference book on these subjects. The importance of accuracy in these matters, if a satisfactory costing system is to be installed, is brought out in the next section dealing with cost accounts.

(b) *Cost Accounts.*—It is not sufficiently recognised that accurate factory costs are essential to success. The result of a business run without these is that the owners do not know how they stand until their final yearly balance-sheet is made out; on the other hand, with an accurate costing system, although they may not know exactly how they are placed until the final balance-sheet is drawn up, they can form a fairly accurate opinion as to the way things are tending all through the year. Such a system to be a success, however, means close co-operation between the business and technical managers and the accountants, and it costs money which, because it does not show direct results, is



often begrudged by short-sighted proprietors. Consequently, Section VI. A. is of vital importance to proprietors, or the financial management, as well as to managers and accountants.

When we come to the section dealing with cost allocation, the peculiar nature of the chemical industry becomes apparent. Yields, which play so vital a part in the chemical factory costs, are not a factor in engineering, and so do not come within the scope of this book, but much useful information will be gleaned from the section on cost allocation, though the succeeding sections on standing orders, departmental or intermediate process accounts, and stock production account will be of limited value or interest to trades other than engineering. The section on oncost or expenses, as we prefer the term, is, on the other hand, of importance to all concerned. The fact that interest on capital and depreciation is a logical part of expenses is emphasised as it should be. Here again, however, the chemical manufacturer will have to apply the information to his own particular needs. To mention one instance, the amount and value of steam used in chemical operations is a considerable part of the expense, but finds no mention or application as applied to engineering costs.

The chapter on works routine—specimen forms—gives a mass of information—150 pages—in the nature of forms which are, however, of little interest to those not engaged in engineering, though useful for the purpose of reference.

Looking at the book as a whole, the impression forced upon the chemical manufacturer, and probably upon all but those engaged in the engineering trade, is that it contains an immense amount of information; that parts of it will prove at once of use to those in other than the engineering industry, and that to those who have the time and patience to read it through carefully there are many pages which, although they deal directly with engineering, will give food for thought and may perhaps give rise to ideas in the particular industry in which the reader is engaged.

The book is well printed on good paper (medium 8vo) and full cloth bound. It is published at the not extravagant price, at the present time, of 45s. net.

H. A. D. JOWETT.

A RESEARCH ON THE EUCALYPTS AND THEIR ESSENTIAL OILS. By RICHARD T. BAKER and HENRY G. SMITH. Second edition. Pp. xv.+471. Published by authority of the Government of the State of New South Wales, Sydney, 1920.

Since the time of Baron von Mueller the systematic study of the Eucalypts has been almost entirely in the hands of Baker and Smith. These authorities have departed from purely orthodox methods in classification and have founded species on a system depending not only on morphological characters, but have called to their aid, *inter alia*, the chemical properties of the products of the trees. The system that has been adopted appears to have worked out very successfully.

In the volume under review, which is enriched with numerous beautiful illustrations, the whole of the important Eucalypts are fully described. Information is given concerning the botanical characters of the tree, its habitat, the character of its timber, and a full account of its essential oil.

A special chapter is devoted to a description of the principal constituents found in the Eucalyptus oils, including cineol, geraniol, the terpineols, piperitol, eudesmol, cuminal, aromadendral, and cryptal. A critical survey of the methods for the determination of cineol in eucalyptus oils is included, but the *ortho*-cresol method has, unfortunately, been published in this country just too late to be included in this survey. The authors

conclude:—(1) That an accurate method for determining the amount of cineol in eucalyptus oils under all conditions has yet to be discovered; (2) that no one present method is applicable in all cases; (3) that arsenic acid is less advantageous for the purpose than phosphoric acid; (4) that the use of petroleum ether in connexion with phosphoric acid is an advantage if the process be carried out in the manner suggested by the authors.

Among other conclusions they agree with most English chemists that the resorcinol method is only of use with certain types of oil. It is stated that eudesmol occurs in both the liquid and the crystalline conditions, "although the line of demarcation separating them is evidently slight." The authors consider that eudesmol changes its character from crystalline to liquid, and *vice versa*. The explanations of this do not appear to be very satisfactory, and it is probable that further research may prove the existence of isomeric eudesmols, each of constant characters and existing in varying proportions naturally, so that superfusion and similar phenomena may be in question.

Cryptal is a new aldehyde of some interest, and was obtained from two different specimens as a crystalline semicarbazone, melting at 180° C.; there appears to be no doubt of its chemical individuality. Piperitone, the "peppermint" ketone discovered by Smith, is another very interesting constituent of certain Eucalyptus oils, and interesting speculations as to its constitution are made. It is possible that thymol, which results from the oxidation of piperitone by means of ferric chloride, may eventually be prepared in this way on a commercial scale.

The work is very free from errors, and reflects great credit on all concerned in its production.

ERNEST J. PARRY.

THE TECHNICAL EXAMINATION OF CRUDE PETROLEUM, PETROLEUM PRODUCTS, AND NATURAL GAS. By W. ALLEN HAMOR and F. WARDE PADGETT. Pp. x.+573. (New York: McGraw-Hill Book Co., Inc. 1920.) Price 30s. net.

This work, primarily written for American students of petroleum technology, will prove of service to all those interested in the examination of petroleum products. The authors have attempted, with success, to compress into a small space the latest physical and chemical methods considered essential for the evaluation of crude petroleum and its derivatives. In order to do this other well-known text-books on the subject have been widely quoted, and the authors have not failed, where they have considered it expedient, to cut out details and satisfy all requirements by referring to the source where such details can be found. Unlike inorganic analysis, the analysis of petroleum bodies depends largely upon empirical methods, carried out according to standard methods in order to obtain comparative results. In such cases the authors have adhered to American methods and have chiefly described those prescribed by the American Society for Testing Materials and the United States Bureau of Standards. For this reason the Redwood viscosimeter is mentioned but not described. The multiplicity of methods now used for reaching the same objective only emphasises the urgent necessity for the early international standardisation of methods in order to save time and prevent confusion.

In addition to chapters on the examination of crude petroleum, illuminating oils, lubricating oils and greases, and bituminous road materials there are valuable chapters on the examination of other petroleum products, the sampling of petroleum oils, the evaluation of oil shales, laboratory methods for benzol-recovery plant operations, and the examination of natural gas. In view of the increasing im-

portance of benzol and the world-wide interest now taken in oil shale, the inclusion of these two chapters adds to the value of the volume. For the latest information on the "Laboratory Testing of Oil Shale for Oil and Ammonia Yield," the paper by Lawson Lomax and Remfry, in the *Journal of the Institution of Petroleum Technologists*, Vol. 7, Jan., 1921, should be consulted.

The descriptive matter in the book occupies circa 300 pages, and the remaining 272 pages are closely packed with an exhaustive appendix containing in compact form chemical and physical data and many valuable specifications for the reference of the refinery chemist, petroleum engineer, and gas engineer. The authors have not failed to give consideration to that very important item in a book of reference—the index—and to emphasise their appreciation of this feature they quote the following from Horace Binney:—"The best book in the world would owe the most to a good index, and the worst book, if it had but a single good thought in it, might be kept alive by it."

The thought underlying the structure of this work is so good that its continued appearance as a standard handbook on the subject in its present form is to be expected. There is no doubt that the analyst or petroleum technologist in possession of this book has at hand a reliable work of reference, and with the methods described and those referred to is in a position to carry out all the essential tests on petroleum products.

M. B. BLACKLER.

## OBITUARY.

J. W. LEITCH.

John Walker Leitch, who died on April 10, was born at Paisley, in 1865, and was educated at the Royal College of Science, South Kensington, and at the University of Glasgow, where he took the B.Sc. degree. In 1887 he came to Milnshridge, near Huddersfield, as secretary and chief chemist to Messrs. Dan Dawson Bros., Ltd., one of the pioneers in the colour-making industry. In 1890 he started business on his own account and shortly afterwards established the present firm of Messrs. John W. Leitch and Co. at Milnshridge Chemical Works. Throughout his career Leitch devoted himself to the manufacture of coal-tar products. Specialising on the nitro- and amino-derivatives of benzene and toluene, he established a standard of excellence in the intermediates and colours which he manufactured that was not bettered even by the Germans. Not only have the effects of his work been felt in the colour industry, but also in the heavy chemical trade and in the field of explosives manufacture, where his intimate knowledge of the aromatic nitro-compounds proved of great value. Leitch held numerous patents covering improvements to processes and to plant, particularly in respect of the recovery of sulphuric acid from waste acid, plants to his specification having been installed in numerous works. Years before the war he developed the manufacture of dinitrotoluene for dye-making, incidentally obtaining TNT, and extended the production of the latter in the early period of the war, producing an almost perfectly pure product. He gave unstintingly of his energy to assist the Authorities in the manufacture of explosives, and as the result of his exertions on war work he suffered a very serious breakdown in health in 1917, which lasted for twelve months, and from the effects of which he never completely recovered.

Leitch was first president of the Chemical Em-

ployers' Federation (Yorkshire Section), an active member of the Association of British Chemical Manufacturers, and was keenly interested in the improvement of facilities for higher technical instruction in Huddersfield, where he was a member of the Technical College Coal-Tar Extension Committee. He was chairman of the Board of Trade Commission that visited the German colour works in the occupied territory, and reported on their condition shortly after the armistice was signed. A member of this Society since 1894, he was until recently a regular attendant at the Society's annual gatherings.

L. G. PAUL.

## PUBLICATIONS RECEIVED.

- FAMOUS CHEMISTS: THE MEN AND THEIR WORK.** By SIR WILLIAM A. TILDEN. Pp. 296. (London: George Routledge and Sons, Ltd.; New York: E. P. Dutton and Co. 1921.) Price 12s. 6d.
- OILS, FATS, AND WAXES AND THEIR MANUFACTURED PRODUCTS.** By C. R. ALDER WRIGHT and C. A. MITCHELL. Third edition, revised and greatly enlarged. Pp. 939. (London: Charles Griffin and Co., Ltd. 1921.) Price 56s.
- TABLES OF REFRACTIVE INDICES. VOL. II. OILS, FATS, AND WAXES.** Compiled by R. KANTHACK, edited by J. N. GOLDSMITH. Pp. 295. (London: Adam Hilger, Ltd. 1921.) Price 25s.
- TECHNICAL METHODS OF ANALYSIS.** By R. C. GRIFFIN. Pp. 666. (New York and London: McGraw-Hill Book Co., Inc. 1921.) Price 33s.
- METALLIC ALLOYS: THEIR STRUCTURE AND CONSTITUTION.** By G. H. GULLIVER. Fourth edition. Pp. 439. (London: Charles Griffin and Co., Ltd. 1921.) Price 15s.
- THE CLAYWORKER'S HAND-BOOK.** By A. B. SEARLE. Third edition, revised, enlarged, and largely rewritten. Pp. 381. (London: Charles Griffin and Co., Ltd. 1921.) Price 21s.
- THE EXTRA PHARMACOPEIA. VOL. II.** By W. H. MARTINDALE and W. W. WESTCOTT. Seventeenth edition. Pp. 688. (London: H. K. Lewis and Co., Ltd. 1921.) Price 17s. 6d.
- LABORATORIES, THEIR PLANNING AND FITTINGS.** By A. E. MUNBY, with a historical introduction by SIR A. E. SHIPLEY. Pp. 220. (London: G. Bell and Sons, Ltd. 1921.) Price 25s.
- DRUGS IN COMMERCE.** By J. HUMPHREY. *Common Commodities and Industries.* Pp. 116. (London: Sir Isaac Pitman and Sons, Ltd. 1921.) Price 3s.
- KOLLOIDCHEMIE DER EIWEISSKÖRPER. PART I.** By PROF. WO. PAULI. Pp. 111. (Dresden and Leipzig: Th. Steinkopff. 1920.) Price: paper, 10 mk.
- DIE PHYSIKALISCHE CHEMIE IN DER INNEREN MEDIZIN.** By PROF. H. SCHABE. Pp. 569. (Dresden and Leipzig: Th. Steinkopff. 1921.) Price: paper, 21s., bound, 23s. 10d.
- FESTSCHRIFT AUS ANLASS DES SIEBENZIGSTEN GEBURTSTAGES VON CORNELIO DOELTER.** Edited by H. LEITMEIER. Pp. 96. (Dresden and Leipzig: Th. Steinkopff. 1921.) Price: paper, 12 mk.
- VORLESUNGEN ÜBER CHEMISCHE TECHNOLOGIE. BAND II, ORGANISCHER TEIL.** By H. WICHELHAUS. Fourth edition, revised and enlarged. Pp. 447. (Dresden and Leipzig: Th. Steinkopff. 1920.) Price: paper, 40 mk., bound, 45 mk.

## AMMONIA OXIDATION AT HÖCHST

J. R. PARTINGTON.

The ammonia oxidation plant of the Höchst Farbwerke (vormals Meister, Lucius und Brüning) is situated at Höchst, on the right bank of the Main, about 10 miles from Mainz. It is about two miles from the works of the Griesheim-Elektron Co., with which, however, it has no connexion. During the war an amalgamation with the Badische interests took place, synthetic ammonia from Oppau being supplied to Höchst for oxidation (*cf. J.*, 1921, 99 n). There was before the war an oxidation plant at Höchst, comprising 37 converters fitted with circular platinum gauzes about 7.2 in. diameter, contained in vessels similar to those used for contact sulphuric acid. The efficiency of this plant, which produced about 54,000 tons of nitric acid per annum, was not above 80 per cent., and a new plant, completed in February, 1919, was erected in its place at a cost of about £1,000,000. This plant has a capacity of 24,000 tons of ammonia, or 140,000 tons of nitric acid, per annum.

In addition to synthetic ammonia, arriving as 25–27 per cent. liquor from Oppau, gas liquor ammonia (20 per cent.  $\text{NH}_3$ ), and cyanamide ammonia liquor (22–27 per cent.) from Knapsack (*cf. J.*, 1919, 303 n) were received, the liquor being stored in four overhead spherical steel tanks, each of 264,200 gallons capacity. The liquor was rectified in four stills, one spare, of the Savalle type with 15 effects, each with a capacity of 20 tons of ammonia daily. The gas not used for oxidation was absorbed in a plate tower 30–40 ft. high. Ammonia from gas liquor was scrubbed with caustic soda solution of 48–53° Tw. and then passed through charcoal purifiers in the usual way to remove sulphur compounds. The ammonia gas from the stills was freed from moisture by refrigeration with ice-cold water; the presence of moisture caused scaling of the iron pipes and was also believed to be prejudicial to the conversion, although there is no evidence of this.

The ammonia gas and air were metered through Venturi meters and driven by ten 55 kw. centrifugal blowers to the filters and converters. The mixture contained 12.5 per cent. by volume of ammonia, regulated within narrow limits. The ammonia supply pipe was 6 in. diameter and the air pipe 18 in. diameter. The air was filtered through cloths in a wooden box and the mixed gas was carefully filtered from dust and ammonium salt fumes through eight layers of linen cloth in chambers 9 ft. by 5 ft.

The gas passed under 6 in. water pressure through two central mains in the converter house, from which it was diverted to the 224 converters, each provided with a circular platinum gauze catalyst of 20 in. diameter, or 2.12 sq. ft. cross section. The maximum output of each converter was 1.5 tons of nitric acid per 24 hours, the efficiency of oxidation being 89 per cent. The efficiency from ammonia to dilute acid (including the efficiency of the absorption plant) was 87 per cent.; that from ammonia to strong acid (including the concentrating plant) was 84 per cent. The body of the converter (*Fig. 1*) consists of cast iron, in four sections with flanges. The two middle sections were 18 in. and 12 in. high, and supported the catalyst between them; the conical top and bottom pieces were each 3 ft. 6 in. high. The top cone was lined with sheet nickel to protect it from the hot gas. The catalyst, clamped between asbestos rings, was started by heating to redness by a hydrogen or coal-gas flame inserted below it through a lighting hole shown in the figure.

The exact construction of the catalyst has been differently stated. It has been asserted that it consists of two, or three, or four platinum gauzes, 20 in.

diameter, of 0.06 mm. wire with 80 meshes to the linear inch. The gauzes are possibly spot-welded and are supported on a grid of ten stout platinum wires, 1 mm. diameter, spaced at 10 cm. distance. The total weight of platinum in the catalyst has been stated as 333 grams, which does not agree very well with the details of construction, but is probably approximately correct.

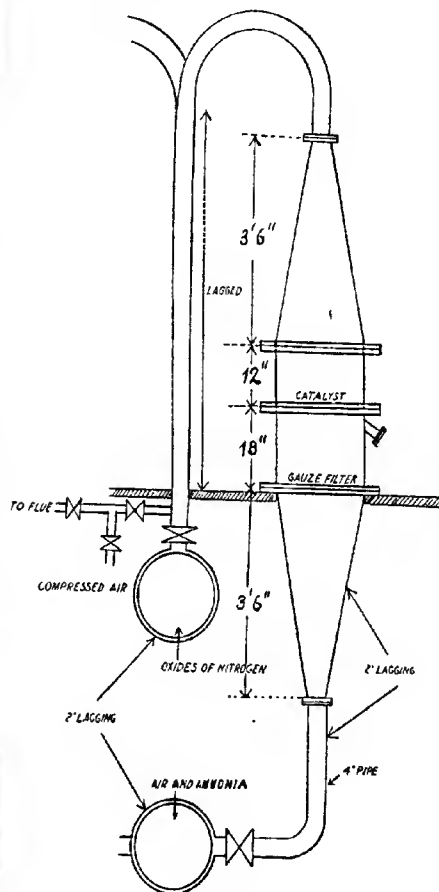


FIG. 1.

The converters were arranged in two sets of seven or eight on each side of separate inlet and outlet mains, the latter 1 ft. in diameter and connected with the converters by 4 in. pipes, lagged with diatomite brick shaped to fit, cemented and wired. The upper bend pipe of the converter was not lagged, so as to allow for expansion. Before entering the converters the gas passed from a common main to a vertical, lagged, multitubular pre-heater, 15 ft. by 5 ft. external dimensions, in which it was heated by steam. The purpose of this was to prevent condensation of acid from the hot gases coming from the converter when these were passed through the heat-exchanger which followed. In this, which was identical in dimensions with the pre-heater, the warmed gas passed in counter-current to the gas from the converters, and left at 200° C. Two heaters so arranged served 14 converters. The temperature of the catalyst was about 800° C.; the gas left the converter at 650–700° C. In starting the converters extra air could be added and the only

M

partly converted exit gas by-passed to a chimney until the gauze was activated. Gate valves were used, with a slight air pressure on the side of the valve not in contact with the gases to prevent leakage. In 56 of the latest type of converters the gas was passed by a lagged 3 ft. main through two tubular boilers, leaving these at 270° C. to the heat exchanger. In this way four tons of water was evaporated per hour and the steam was used for warming the gas, as described above. From one to three men only are necessary to attend to all the converters.

The gases containing nitric oxide and steam coming from the converters were passed through eight cast-iron mains 18 in. diameter, supported on three ferro-concrete bridges, to four rows of eight absorption towers. Each row of towers was provided with twelve intensive coolers of special design, i.e., 48 coolers in all. These coolers (Fig. 2) were of aluminium, with an annular space between the outer wall and an inner vessel, the gas entering above through a 4-in. inlet, dividing into four 2-in. pipes leading to an annular space between the walls. The gas and condensate left through a 4-in. outlet below. The coolers are 5 ft. high and 2 ft. 6 in. diameter. In these, on account of the rapidity with which the gas passed through, practically no acid was condensed, but only water. The condensate was put down the fifth absorption tower. The gas left the set of 4 coolers at 30° C. through 4 pottery mains 1 ft. diameter, joining to a single brick chamber at the base of each set of eight towers attached to 56 converters. Secondary air was admitted at this point (not before the coolers).

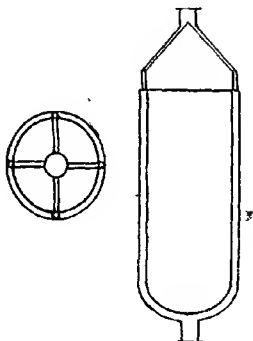


FIG. 2.

The towers are 41 ft. high and 21 ft. diameter internally. The walls comprise three layers of brickwork, the outer layer of bricks 6 in. by 4 in., set in a mixture of sodium silicate and kieselgub. The top of the tower is slightly domed, and there is a central partition in each tower, the gas passing up one side and down the other, and then going to the next tower through a brick channel at the base. The towers are packed with stoneware rings. The total internal volume of all the towers is 35,320 cb. ft., which amounts to only 81 cb. ft. per lb. NO<sub>2</sub> absorbed per minute as compared with 350 cb. ft. calculated by the formula proposed by Dr. Parker and the writer (*cf. J., 1919, 75 r*), or 1500 cb. ft. as used at the French works at Bassens when we saw it in 1918. The reason why this small tower capacity is effective is clear when it is remembered that at Höchst no less than 25 per cent. of the oxides was absorbed as nitrate by saturated sodium carbonate solution fed to the last two towers. The liquor in the first of these towers is allowed to become acid, so that the nitrite is converted into nitrate, whilst that in the second is kept

alkaline, to absorb the oxides of nitrogen driven out of the first when conversion into nitrate occurs (see B.P. 129,699/1919; Partington and Rideal). The solution of nitrate was evaporated by the waste heat of the converters. The remaining towers were fed with water for the production of nitric acid. The efficiency of absorption plant was 97 per cent. Each tower is stated to have cost 40,000 marks. Acid of 50 per cent. strength was obtained from the first tower of the series, the liquid being circulated in the towers by 8 in. chrome steel centrifugal pumps each fed by a 3-4 in. pottery main and delivering through a silicon-iron pipe of the same diameter to the top of the tower, two delivery pipes being used to each tower. The metal pipes were coned and fitted in the same way as the pottery pipes. Acid distribution was effected at the top of the tower by a revolving ribbed disc, turned by a gear shaft at 80 revs. per min.

The acid from the towers was passed to the coolers, consisting of 8-pipes of silicon-iron cooled externally with water. There were also intercoolers between each pair of towers except the last, and the temperature was kept as low as possible, 25° C. in winter and 40° C. in summer. The fact that the dilute acid towers would probably work slightly better if warm (see Partington and Parker, *loc. cit.*) does not seem to have been appreciated. In summer ice-water was used in the coolers. Large stoneware receivers were placed above the coolers to serve as acid reservoirs. The circulation in each tower was 110 gallons per min.; the loss of acid in the whole system did not exceed 2 per cent.

Twelve stock tanks of brickwork, each holding 1412 cb. ft., received the tower acid from the receivers at the base of the towers, through tapered pottery pipes. No fan was used with the towers.

The tower acid was concentrated by mixing 7 parts of 50 per cent. tower acid with 1 part of sulphuric acid, in five mixing vessels with mechanical stirrers and then denitrating. About 42,000 tons of sulphuric acid was used per month, but practically all was recovered. (In France 2.5 to 3 parts of sulphuric acid only were used, but the nitric acid obtained was less concentrated—85 per cent. strength—than that obtained at Höchst. The nitric acid was recovered partly in two rows of nine silicon-iron towers, 4 ft. diameter and 25 ft. high, and partly in stone towers, with cast-iron pipe jackets, 36 ft. high and 3 ft. 6 in. external diameter, the walls being 9 in. thick. Steam at 150° C. was introduced at the base by a 1 in. lead pipe just above the exit for the denitrated acid. The nitric acid vapour from the top of the denitrator passed to a cooler of S-shaped silicon-iron pipes. The acid to be denitrated entered half-way down the cooler and acid from stock was sent down the upper part to assist condensation. Oxides of nitrogen were removed from the nitric acid by a current of air and recovered in 20 absorption towers. The completely denitrated sulphuric acid was cooled in a water-cooled cast-iron cooler and collected in a large lead-lined tank of artificial stone. It contained about 70 per cent. sulphuric acid and was reconcentrated in a Kessler apparatus or in a modified Pauling apparatus (*cf. J., 1920, 656 A*). The latter was said to be very efficient. (In French practice a denitrating column 20 ft. high and 18 in. internal diameter produced 6 to 8 tons of nitric acid per day with an overall yield of 85 to 87 per cent. on the ammonia burnt in the Ostwald type catalysts. The consumption of fuel in reconcentrating the sulphuric acid was 15 to 20 per cent. of the weight of nitric acid produced.)

It is of interest to compare the mode of operation at Höchst, representing the latest German practice, with the indications obtained by experimental work in England, unfortunately never translated into technical practice. The resemblance is striking. In the following comparison the experiments carried

out by the writer and his colleagues for the Munitions Inventions Department are referred to, many of which have not been published. The separation of water from the converter gas by rapid cooling was the subject of B.P. 131,942/1918 (Rideal and Partington). The object of this procedure, which is combined with the addition of secondary air *after* the coolers, is to keep the gas as concentrated as possible in the oxidation and absorption towers. It is clear from the equation  $4\text{NH}_3 + 5\text{O}_2 = 4\text{NO} + 6\text{H}_2\text{O}$ , that the uncooled converter gas contains nitric oxide and steam in the ratio 2 to 3 by volume, so that if the steam is removed a considerable concentration in oxides of nitrogen results.

The dimension of the platinum wire and the mesh of the gauze are also those found most suitable by the writer, and if the use of two gauzes at Höchst is the correct description this is also in agreement with the type of catalyst recommended in the official publication, "Ammonia Oxidation applied to Vitriol Chamber Plants," M.I.D., Stationery Office, 1919. The use of alkali to finish the absorption was carried out exactly in the manner specified in B.P. 129,699/1919 (Rideal and Partington); it has been shown that this effected a considerable economy of tower space. The use of sulphuric acid as an absorbent, which was tried at Leverkusen and found unsatisfactory, was also the subject of a long series of experiments (unpublished) by the writer and Dr. Parker, who decided that it was not an efficient method of absorption.

It may fairly be said that laboratory workers in England and Germany seem to have been led to the same conclusions, but whereas the German technologists translated the results into plant the English work finished in the research laboratory.

## YEAST GROWTH.

LEWIS EYNON.

The growth of yeast, first closely investigated by Pasteur, has since been the subject of much research and controversy. In suitable media, such as malt wort, the growth is characterised by three phases, viz.:—(1) the quiescent, or lag phase, observed when old yeast cultures are introduced into fresh wort, (2) the logarithmic phase or period of unrestricted growth, and (3) the phase of retarded growth due to exhaustion of oxygen or food, or to accumulation of carbon dioxide and other toxic substances. The yeast crop finally obtained is obviously dependent on the various factors which retard growth. It is shown by Dr. A. Sclator (Chem. Soc. Trans., 1921, 119, 115) that if yeast is grown under such conditions that only one of these factors comes into play, it is possible to determine the influence of the individual factors on the yeast crop. Thus, provided the ratio of the constant of growth,  $K$ , to the fermentative activity of the yeast,  $F$ , is constant, the yeast crop should be approximately proportional to the initial concentration of the sugar when this constituent of the medium is made the limiting factor (cf. J., 1919, 392R). The proportionality was tested with a wild yeast of the *S. ellipsoideus* type grown in a fermented wort that had been freed from alcohol and treated with small quantities of glucose; direct proportionality between yeast crop and initial glucose concentration was found to hold up to about 1 per cent. of sugar, 3900  $\times 10^4$  cells being produced per 1 gram of sugar fermented. It is further found that with sugar as the factor controlling growth the yeast crop (*S. cerevisiae* grown in wort) is practically independent of the fermentation temperature between 10° and 25° C., as should be the case since the temperature coefficients of  $K$  and  $F$

do not differ greatly. The influence of the amount of seeding on the yeast crop is nil provided that actively growing cells are used for seeding, i.e., the crop is the sum of the seeding and the growth, the latter being constant. The retarding influence on growth of carbon dioxide remaining dissolved in the wort is very marked and is not attributable merely to exclusion of oxygen. With regard to the influence of oxygen on yeast growth, Sclator has attempted to verify the opinion that malt wort contains a certain amount of combined oxygen available for yeast growth (cf. J., 1919, 392R) by estimating yeast crops under such conditions that the oxygen-containing substance would become the limiting factor determining the crop. His experiments, however, do not bear out this opinion, but suggest that there are two different modes of growth of yeast, one of which is illustrated by the initial stages of growth in malt wort, and the other by growth in lactose-yeast-water. In the former, oxygen (either free, combined, or absorbed by the yeast) plays no part save that in the free state it slightly retards growth, carbon dioxide retards growth, and the energy necessary for growth is obtained from the fermentation of the sugar. In the latter mode of growth, which is unaccompanied by alcoholic fermentation, free oxygen is essential, carbon dioxide in large amount retards growth, and the energy necessary for growth is obtained through some oxidation reaction carried out by the yeast.

As Sclator observes, there is some similarity between the views now advanced by himself and those expressed by Pasteur with regard to the influence of oxygen on yeast growth. Thus, Pasteur observed (*Études sur la Bière*) that yeast can live and multiply in absence of oxygen, although under these conditions the amount of yeast produced is very small by comparison with the amount of sugar fermented. Thus, in one experiment in which fermentation was effected with exclusion of air the ratio of the weight of (dried) yeast to that of sugar (sucrose) fermented was 1:89, whilst in another experiment in which a more mature yeast culture was used for seeding, the ratio was 1:176, and the yeast crop showed abundant evidence of involution. Pasteur inferred that in order to grow in absence of oxygen, the yeast cells must be young and vigorous and still under the influence of the vital activity produced by the free oxygen which has aided their formation, and which perhaps they have temporarily accumulated; very old yeast cultures were found to remain quite inert in a medium from which free oxygen was excluded. In further experiments, Pasteur showed that the ratio of yeast produced to sugar fermented or consumed is greatly increased by the presence of air, this ratio being as high as 1:4 under extremely favourable conditions of air access. Under such conditions a considerable part of the carbon dioxide evolved is produced, not by fermentation, but by oxidation resulting from assimilation of atmospheric oxygen, i.e., to this extent the yeast behaves like an aerobic organism. Pasteur subsequently showed that with air access yeast can multiply in a solution of lactose in yeast-water without producing any fermentation whatever, thus proving that yeast is capable of a strictly aerobic existence. He concluded that the fermentative power of yeast can vary widely between limits which are determined by a maximum and a minimum access of oxygen. With maximum access of oxygen yeast ceases to be a ferment, i.e., the ratio of the yeast crop to the weight of sugar consumed is of the same order as for aerobic organisms, such as moulds. This mode of life corresponds with the second mode of growth (growth in lactose-yeast-water in presence of oxygen) outlined by Sclator. With minimum access of oxygen yeast still grows, though much less actively than in presence of more oxygen, and its capacity for fermenting sugar is greatest. This mode of life ("*la vie sans air*")

corresponds to some extent with the first mode of growth (initial stages of growth in malt wort) outlined by Sclator, but Pasteur's view differs from that of Sclator in that the former assigned an all-important rôle to the oxygen with which the yeast has previously been in contact. Sclator considers that the increase in yeast crop obtained by aerating fermenting wort is due, not to the influence of oxygen, but to displacement of carbon dioxide, and he suggests that the larger yeast crop obtained from wort saturated with air compared with that from wort containing no air may be due to lessened super-saturation of the wort with carbon dioxide.

With regard to the two modes of growth, Sclator states that "the factors determining which mode of growth takes place when both are possible have not yet been completely investigated." It is clear, from Pasteur's writings (*loc. cit.*, p. 252), that he considered that yeast can grow in both modes simultaneously, i.e., as a fermenting organism and as an aerobe, and that the predominance of its fermentative or aerobic character depends on the amount of air at its disposal.

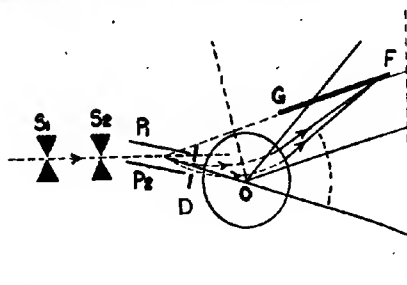
## MASS SPECTRA AND ATOMIC WEIGHTS.

The atomic theory of matter put forward by Dalton in 1803 has remained one of the articles of faith of chemists until very recent years. Even now, only in one of the five main postulates of the theory has a flaw been detected. Dr. F. W. Aston, in a lecture delivered recently before the Chemical Society, reviewed the evidence, largely the outcome of his own work, which has undermined the chemist's and physicist's confidence in the postulate that "atoms of the same element are similar to one another and equal in weight."

The phenomena of radio-activity led to the conclusion that varieties of certain transformation products, more especially varieties of lead, were possible, which, while possessing chemical properties identical in every respect, nevertheless differed in atomic weight. This conclusion was abundantly verified by determinations of the atomic weight of specimens of lead derived from various sources, and the name "isotopes" was suggested by Soddy for such elements which, although exhibiting different atomic weights, yet occupy the same position in the Periodic Table. Moseley's work showed that the chemical properties of an element depend not on its atomic weight, but on its atomic number, which connotes the number of units of positive electricity on the atomic nucleus. Thus the atomic number of hydrogen is 1, of helium 2, of lithium 3, and so on. J. J. Thomson, by the method of positive-ray analysis, obtained evidence that neon, of which the accepted atomic weight was 20.20, was really a mixture of isotopes of atomic weights 20 and 22. The evidence was not as sufficiently conclusive as desired, and Aston undertook further investigations, using Thomson's method, but varying the procedure in order to obtain greater accuracy.

The apparatus employed is illustrated in the accompanying figure.\* Positive rays proceeding from the perforated cathode of a cathode-discharge tube are sorted out into a thin strip by means of two parallel slits,  $S_1S_2$ , and are then spread into an electric spectrum by means of the charged plates,  $P_1P_2$ . A portion of this spectrum is isolated by the diaphragm  $D$  and passed between the circular poles of a powerful electromagnet  $O$ , whereby the rays are bent back through an angle

more than twice the initial deflection. As a result, rays constituted of corpuscles having a constant mass converge to a focus, and a spectrum dependent upon mass alone may be photographed on a plate placed at  $GF$ .



The measurements of mass made in this way are relative to lines of known masses, thus  $O$ , 12;  $CH$ , 13;  $CH_2$ , 14;  $CH_3$ , 15;  $CH_4$ , 16;  $CO$ , 28. When neon was employed in the tubes, spectrum lines corresponding to masses 10, 11, 20 and 22 were obtained. The first pair are what are termed second order lines, and arise from the presence of rays carrying a double charge. The relative intensities of the remaining lines indicated that neon was a mixture of an isotope of mass 20 with 10 per cent. of an isotope of mass 22, giving an average atomic weight of 20.20 in accordance with the accepted figure. The method has been applied to a number of electronegative elements, and lately it has been found possible to extend the method to the investigation of electropositive elements. The results obtained are summarised below:—

Table of Elements and Isotopes.

Element.	Atomic number.	Atomic weight.	Minimum number of isotopes.	Masses of isotopes in order of intensity.
H	1	1.008	1	1.008
He	2	3.99	1	4
B	5	10.9	2	11, 10
C	6	12.00	1	12
N	7	14.01	1	14
O	8	16.00	1	16
F	9	19.00	1	19
Ne	10	20.20	2	20, 22, (21)
Si	14	28.3	2	28, 29, (30)
P	15	31.04	1	31
S	16	32.08	1	32
Cl	17	35.46	2	35, 37, (39)
A	18	39.98	(2)	40, (50)
As	33	74.96	1	75
Br	35	79.92	2	79, 81
Kr	36	83.92	8	84, 86, 82, 83, 80, 78
I	53	126.92	1	127
X	54	130.2	5, (7)	129, 132, 131, 134, 135, (128, 130?)
Hg	80	200.8	(6)	(197-200), 202, 204
Li	3	6.94	—	6, 7
Na	11	23.00	1	23
K	19	39.10	2	39, 41
Rb	37	85.46	2	85, 87
Cs	55	132.81	1 (?)	133

The numbers in brackets are provisional. The results indicate that within an accuracy of 1 in 1000, the weights of the atoms of all the elements measured, except that of hydrogen, are whole numbers, thus re-establishing Prout's hypothesis, but with the modification that the primordial atoms are of two kinds, viz., the electron, or atom of negative electricity, and proton, the atom of positive electricity. A neutral atom of an element of atomic number  $N$  has a nucleus consisting of  $K+N$  protons and  $K$  electrons, and round this are set  $N$  electrons. An atom of atomic weight  $m$  is turned into one of atomic weight  $m+1$  by the addition of a proton plus

\* Reproduced, by permission, from the *Philosophical Magazine*.



an electron. If both enter the nucleus, the atomic number is unaltered, and an isotope of the original atom results. If the proton alone enters the nucleus, an atom of the next higher atomic number results. If both new configurations are possible, elements of the same atomic weight but with different chemical properties result. Such elements are called "isobares." Their occurrence among radioactive elements is known. The hydrogen atom is unique; it is the only atom in which the nucleus is not composed of a number of protons packed very closely together. When four protons are packed closely with two electrons to form the helium nucleus, theory indicates a reduction of the effective mass, so that the helium nucleus will have a weight somewhat less than four times that of hydrogen. The lecturer concluded with the remark:—"Though as a chemist I view with some dismay the possibility of eighteen different mercuric chlorides, as a physicist, it is a great relief to find that Nature employs at least approximately standard bricks in her operations of element building."

## SOCIETY OF CHEMICAL INDUSTRY.

### NEWS FROM THE SECTIONS.

#### SHAWINIGAN FALLS.

A dinner was held by the Shawinigan Falls Section at the Cascade Inn, Shawinigan Falls, on April 12, and was attended by about forty members.

After the customary toast to the King, the chairman, Dr. F. W. Skirrow, introduced Mr. V. C. Bartram, of the Canadian Electro-Products Co., who gave a very interesting address on the situation in Europe. Mr. Bartram passed a considerable portion of the year 1920 travelling in England, Holland, Belgium, France, Italy, and Switzerland, and he spent a month in Germany visiting most of the larger chemical centres. He was much impressed by the difference between the commercial conditions prevailing in Germany and those in England. There seemed to be absolutely no unemployment in Germany, and most industries were working at full capacity, especially those industries which were able to obtain their raw materials in the country. In many instances, however, plants were working at full capacity on raw materials obtained through outside interests, the owners of the materials being charged so much per kilogram or metre for the work done upon them. In this way employment was being maintained in Germany and unemployment in surrounding countries. Unless steps were taken to protect industries in Allied and neighbouring countries, it would, in Mr. Bartram's opinion, be very difficult for their manufacturers to compete with German manufacturers for some considerable period. Mr. Bartram found the German people very polite, and anxious to impress one with the extent of their wealth. It would appear as if the Germans, not knowing the exact steps which are going to be taken to collect the indemnity, are very keen to dispose of such wealth which they have retained in Germany to their own personal advantage, i.e., to get the most enjoyment out of the money while they have it. Mr. Bartram is of the opinion that by proper control of industry and increased taxation, the Germans could well afford to pay the indemnity demanded by the Allies, and that at the present time they are really prosperous, although very short of some of the real necessities of life; however, they seemed to be reconciled to getting along with a minimum of luxury and without many of the things we here consider necessities. Owing chiefly to the enormous inflation of money, a traveller in Germany gains the impression that

the country is immensely rich, and that there are only two classes in Germany to-day, the extremely rich and the extremely poor. The inflation of money has been much greater than the increase in prices, and many persons who had property to dispose of have sold it and become very rich almost without effort. It would appear that Germany can only hold her present advantages in many of the world's markets she is now dominating until such time as the German populace tires of living under the conditions existing there to-day. There is no doubt that the Germans realise they have been beaten, but many feel that by sticking closely to work and doing everything possible to maintain production on an economic basis they are dealing a severe blow to Allied interests, and in this way getting back some of their own. It would hardly seem possible that the German populace can long continue to maintain their present standard of living, and doubtless the trade situation will rapidly be forced down to a level at which Germany will cease to enjoy her present advantages. The import duties adopted by other Governments to counteract the advantages Germany now enjoys through exchange should improve the situation in the rest of the world and particularly within the Empire.

Mr. Vinet, of the Shawinigan Water and Power Co., then made a few remarks on his visit to Germany with the Allied Army of Occupation in 1919.

#### MANCHESTER.

About 70 members attended the last meeting of the Session, which was held at the Textile Institute on May 6, Mr. John Allan presiding.

In a short communication on "Woad," Mr. J. Hühner stated that the prohibition of the import of dyes could be traced back at least to the 15th century, for he had found in the British Museum a special licence granted and signed by Henry VII. for the importation of 230 tons of woad from France. Although woad grew in this country from very early times, it was not cultivated here until the 15th century. When serious attempts were made to cultivate it, its importation was prohibited. Many of these attempts failed because continental growers, although willing to supply seeds, destroyed their germinating power by boiling them in water or smoking them in chimneys.

Mr. W. H. H. Norris read a paper on "Methods for the Complete Recovery of Nitro Cake," in which he described a process which had been used on a manufacturing scale. This consists, briefly, in digesting the cake with hot process liquor and so precipitating all the salt cake in a pure state, leaving the acid in solution. The mixture is filtered and washed in centrifugals, and the resulting acid liquor is used for ammonia absorption. The ammonium sulphate liquor is evaporated until a definite density is reached, and the process liquor is run back to the digester to neutralise the acidity of a fresh charge of nitro cake.

A new process for recovering sodium sulphate and a strongly acid liquor was also described, which involves running off the residue from the retort and pelleting it in a digester in presence of liquors of regulated composition. A second treatment of the liquor in the same vessel produces anhydrous sodium sulphate (96 per cent.). For transport, the acid liquor of 60 per cent. acidity may be diluted to about 25 per cent. acidity without risk of crystallisation taking place when cooled to 10° C.

Mr. R. Curtis, in a paper on "Works Accounts from the Chemist's Point of View," referred to the excellent work carried out during the war by the Department of Explosives Supply, and illustrated the type of system required by the works chemist with particulars of the method used at H.M. Factory, Avonmouth. The works chemist needs a detailed analysis of all manufacturing costs which must be made available at the earliest possible

moment. Costing information supplied two or three months after the expenditure has been incurred is of little value.

#### ANNUAL MEETING, 1921.

##### CANADIAN ASBESTOS.

The asbestos-mining companies of Quebec have extended a cordial invitation to the members of the Society to visit their mines and mills on the occasion of the Annual Meeting in Montreal.

Although 85 per cent. of the world's asbestos is produced in Canada, it all comes from a comparatively small district about three hours' journey from Montreal. There are fourteen principal producers located at Coleraine, Black Lake, Thetford, East Broughton, and Danville.

The mining and milling of asbestos consists of quarrying proper, and the "cobbing" or separation of the fibre from the rock.

The first operation in opening a quarry is the removal of the surface soil, usually from 5 to 25 feet deep, for which steam shovels are employed. The exposed rock is drilled by hand, machine, or electric percussion-drills. This is followed by blasting with dynamite, every pound of which brings down about five tons of rock. Hand-cobbing separates the crude material into three grades:—(a) The long asbestos fibres and pieces of rock containing them; (b) the milling material or rock containing the shorter fibres; and (c) barren rock which goes direct to the dump.

The material specified in (a) is sent direct to the cobbing sheds, where the fibres are separated into different grades both by hand and machinery. The rock containing the shorter fibres is crushed and the asbestos separated from the waste by exhaust fans. The asbestos is now ready for the market, and is classed into two grades, termed respectively the "crude" and the "fibre." The crude is divided into two grades, No. 1 having fibres over 0.5 in. long, and No. 2 containing fibre under 0.5 in. These grades are used for asbestos cloth, pipe-covering, and other articles of which asbestos is the principal constituent. The material of shorter fibre is used in other manufactures, chiefly as a filler.

The production of asbestos has grown from 1400 tons, valued at £30,000, in 1885, to 120,000 tons, valued at £2,250,000, in 1919.

#### MEETINGS OF OTHER SOCIETIES.

##### THE INSTITUTE OF METALS.

On May 4 Prof. T. Turner, of Birmingham University, delivered the Eleventh May Lecture, on "The Casting of Metals." Sir George Goodwin, president of the Institute, was in the chair.

In opening, Prof. Turner referred to the castings of antiquity, mainly bronze, silver, and gold, many of which were of high quality and great beauty, but there was no reason to believe that the achievements of the past could not be equalled in design and excellence by modern methods, whilst output had been increased enormously and the percentage of wasters reduced. Casting may be defined simply as "the art of filling a given space by means of a molten metal or alloy"; yet it is the exception rather than the rule to obtain a perfect casting. In general, the higher the melting point of the metal or alloy the greater the casting difficulties, partly owing to the condition of the metal, and partly on account of the necessity for obtaining high-class refractories, sands, cores, fuels, etc. A valuable property of metals is their low viscosity in the liquid state; in most cases the viscosity does not exceed appreciably that of water, so that very intricate shapes can be

cast. In addition, the surface tension as a rule is high, and coarse-grained sand moulds can be used since they are not "wetted" by the molten metal or alloy. The latent heat of metals, which governs the rate of solidification, is usually the greater the higher the melting-point, but aluminium is a notable exception, having a very high latent heat. The coefficient of expansion of aluminium is also very high and renders the metal liable to fracture while cooling in the mould. Molten metal, melted in ordinary furnaces, practically always contains in solution gases derived from the atmosphere of the furnace, and these gases may be evolved during solidification in the mould with the formation of blowholes. For most metals and alloys, however, there is a suitable pouring temperature at which the evolution of gas during casting is not excessive. The amount of "superheat" (heating above melting point) which is necessary to obtain sound castings varies greatly with different alloys.

Pure metals, as a general rule, possess the same density whether slowly or quickly cooled. In the case of alloys, especially those in which there is a wide temperature-interval between the liquidus and solidus, slowly cooled castings, such as sand-castings, frequently expand during solidification, and the result of this expansion is reflected in a reduction in the density of the alloy. The work of Prof. Turner and his students, in which the volume changes in a number of typical alloy systems during solidification were investigated, has shown the connexion between the constitution of the alloys and the alteration in volume, and the results can be correlated with the densities of the solid alloys. The lecturer closed with a reference to the causes of failure in casting metals, among which may be mentioned, pouring at an unsuitable temperature, imperfect cores, faults in moulding, cracks, misruns or run-outs, and breakage in handling. The losses from these and other causes are considerable; they usually amount to about 10 per cent. but sometimes to 40 per cent. of the output.

##### THE IRON AND STEEL INSTITUTE.

The annual meeting was held on May 5 and 6 at the Institution of Civil Engineers under the presidency of Dr. J. E. Stead. After the formal business was concluded, Mr. Harry Brearley read a paper on the welding of steel in relation to the occurrence of pipes, blow-holes, and segregates in ingots. His experiments were designed to ascertain to what extent the surfaces of cavities in ingots were welded up in operations such as forging, rolling, etc. He found that welding was as a rule not perfect, although it was frequently impossible to detect the weld in polished and etched samples. Under the notched-bar impact test, however, splitting frequently occurred along the welded surface when this was longitudinal in the test-piece. This tendency to splitting was greater the tougher the steel, and it was suggested that the quality of a weld could be measured by ascertaining the degree of toughness which must be induced in the steel in order to pull apart the welded surfaces in the ordinary Izod test. Mr. S. H. Fowles presented a paper on the cleaning of blast-furnace gas, and gave a description of the Halberg-Beth dry gas-cleaning plant. He pointed out that waste gas and heat should be used with the maximum efficiency, and that to use the gas under boilers was a barbarous practice. He advocated its use in internal-combustion engines, after careful purification in a suitable cleaning-plant; and he pointed out the defects of existing types of gas-cleaner, whether they were based on the wet process, the dry process, or the electrostatic process. Mr. J. E. Fletcher described graphic methods of representing the composition and melting points of various blast

furnace and cupola slags, with a view to obtaining a clearer understanding of the theory of slags and of their practical utility.

The prevention of hardening cracks in a tungsten tool-steel containing 1.1 per cent. carbon and 0.8 per cent. tungsten was discussed by Mr. S. N. Brayshaw in a very long paper which embodied an enormous amount of experimental work. A series of milling cutters of complicated design was used for his experiments, which sought to correlate the liability to crack with the heat-treatment before and during hardening. A paper by Messrs. K. Honda, T. Matsushita, and S. Idei discussed the cause of quenching cracks. These are attributed to structural differences in the quenched samples; in small samples a core of martensite exerts a large tangential tension on the periphery, which consists largely of austenite; cracks in large samples may be formed as a result of stresses due to the structural differences between the inner and outer portions—pearlite and austenite—just below the A1 point. Mr. J. H. Whiteley described a preliminary investigation on the cupric etching effects produced by phosphorus and oxygen, respectively, in iron. From his results it appears that differences in the phosphorus content of less than 0.02 per cent. in adjacent parts of otherwise pure iron can be readily discovered by means of the cupric reagents; as the difference is increased, at least up to 0.15 per cent., the contrast becomes more and more pronounced. On the other hand, attempts have failed to produce an unequal distribution of oxygen which could be detected by cupric reagents in the same manner as that of phosphorus. Two methods of obtaining an unequal oxygen content were used:—(a) By soaking pure iron in its own oxide at a high temperature and then welding it with the unoxidised material; (b) by reducing highly oxidised iron in hydrogen and then welding it with the original metal at a low temperature. On etching, white resist-lines were formed at the weld junctions when oxygen was present, but these resist-lines were not formed when unoxidised iron was welded in dry hydrogen. Dr. Stead also contributed a paper dealing with the question of the solid solution of oxygen in iron, and from his results it appears that the absorption of oxygen into solid solution by iron can no longer be doubted. When iron, which has been heated in air or oxidising gases and has absorbed oxygen, is super-saturated, the oxide falls out of solution in the form of globules of free oxide. These globules become larger and larger as oxidation proceeds, eventually joining together to form continuous layers. Other globules which appear in iron as the result of gaseous action are sulphide of manganese and possibly protoxide of manganese, though in what form the sulphur enters the iron is not known.

Dr. A. Westgren's paper on "Röntgen Spectrographic Investigations of Iron and Steel" was introduced by Sir William Bragg. The paper is a brief report on the first results obtained by the application of Röntgen spectrographic methods to metallographic problems. The work described verifies Hull's result that iron at ordinary temperatures ( $\alpha$  iron) has a cube-centred cubic lattice structure. The same orientation is found at 800° to 830° C., that is, within the so-called  $\beta$  iron interval, from which Dr. Westgren concludes that iron does not undergo any allotropic transformation at 768° C. The crystal lattice of austenite and of pure iron at 1000° C. is different from that of iron at the ordinary temperature. In martensite the iron atoms are grouped in exactly the same way as in  $\alpha$  iron.

Further papers, which were not discussed, included "The Protection of Iron with Paint," by J. N. Friend, "Slip-Lines and Twinning in Electro-deposited Iron," by W. E. Hughes, "The Scientific Control of Combustion," by H. T. Ringrose, and "Comparison of Different Methods of Estimating Sulphur in Steel," by T. E. Rooney.

### ROYAL PHOTOGRAPHIC SOCIETY.

At the meeting held on April 26 Mr. Chapman Jones gave a memorial lecture on Sir W. de W. Abney, one of the best-known workers in scientific photography, president of the Society in 1892—1894, 1896, and 1903—1905, and for many years the Honorary Editor of the Society's Journal. Mr. Jones prefaced his lecture with a short account of Abney's work and interests other than photographic. He was artist, archaeologist, and astronomer, and, more particularly, an organiser and missionary in the application of scientific method to teaching. He was for many years associated with the Science and Art Department, and had considerable success in urging the necessity for adding practical work to class teaching. His researches in photography and allied subjects covered a very wide field. He was the first to apply spectroscopy to photographic research, and closely associated with this was his work on colour vision, colour measurement, and the principles of three-colour photography. He studied also the continuing action of light on bichromated gelatin, various problems concerned with photographic development, the efficiency and speed of shutters, and sensitometry. His work on emulsions included the production of plates specially sensitive to the infra-red, a problem which he studied both from the standpoint of the addition of materials to "weight the silver bromide molecule" and from that of the formation of a blue emulsion with strong absorption in the far-red. Both in this and in other work he several times anticipated modern research, having noticed, for instance, red-sensitising by treatment of plates with sodium sulphite, and having made use of wedge screens, both of silver and of platinum burned into the glass, in density measurements. Abney's activities were so numerous that the lecturer had time for very little more than to indicate their scope, the work on red-sensitive emulsions being treated in more detail; the printed lecture will include a bibliography.

### ROYAL SOCIETY OF ARTS.

In a paper on "Paper Pulp Supplies from India" read before the Indian Section on May 3, Mr. W. Raitt discussed the possibilities of bamboo as the paper-making material of the future, having regard to the fact that the available supplies of timber for wood-pulp manufacture are rapidly becoming exhausted.

The present world consumption of paper may be put at 12 million tons per annum, 80 per cent. of which is made from wood pulp. It is estimated that India can produce annually 10 million tons of bamboo pulp and 3 million tons of pulp from Savannah grasses, and could therefore supply the whole world. Care and experience are necessary to avoid too frequent cropping of the bamboo, whereby its reproductive powers are impaired, but making allowances for these enforced rest periods, a 20,000-acre reserve should keep a factory with an output of 10,000 tons per annum running indefinitely.

Hitherto the chief difficulty in dealing with bamboo has been the high bleach-consumption of the boiled pulp. This may be avoided by modifying the conditions of boiling so that the starchy bodies and pectoses, which are mainly responsible for staining the pulp, are removed before the protective coating of lignin is removed from cellulose. This may be effected by subjecting the bamboo to a preliminary digestion with 1 or 2 per cent. soda at a temperature not exceeding 120° C. This treatment removes both starch products and pectoses without attacking the lignin. Further digestion with 4 per cent. soda at temperatures above 130° C. removes the lignin, leaving a pulp which bleaches comparatively easily. By this method of fractional

digestion, the soda figure is reduced from 26 to 19 per cent., and the consumption of bleach of the boiled pulp drops from 16 to 11 per cent. The sulphate process of boiling can be modified in a similar way, but offers no advantages over the soda process. The treatment of the bamboo before boiling may consist in chipping or crushing. The latter method, though producing a more bulky charge, makes for better penetration and therefore better digestion.

It is estimated that bamboo could be transported to manufacturing sites in Burma at a cost of 12s. 6d. to 15s. per ton, equivalent to £1 11s. 3d. to £1 17s. 6d. per ton of unbleached pulp; the cost of manufacture would be under £10 per ton of pulp, so that taking £4 per ton as the future normal freightage, unbleached bamboo pulp could be delivered in this country for £16—£18 per ton, a figure which should compare very favourably with that of mechanical wood pulp. These figures are based upon estimated future costs of production when the present inflation of prices has passed.

#### THE FARADAY SOCIETY.

A meeting of the Faraday Society was held on May 9, Prof. A. W. Porter presiding. Mr. U. R. Evans introduced a paper by Dr. E. K. Rideal and himself on the Problem of the Fuel Cell, which dealt with attempts by the authors and others to obtain electrical energy by utilising carbon, directly as coal, charcoal, etc., or indirectly in the form of its combustion or reaction products, as one of the elements of a primary cell.

Fuel cells may be classified under three headings:

—(1) Direct fuel cells burning solid fuel, which suffer from current polarisation due to the low velocity with which carbon enters into electrodic reactions. (2) Semi-direct fuel cells using gaseous substances derived from coal, etc., which are used to saturate the negative electrode; these also suffer from current polarisation owing to the difficulty of keeping the electrode material saturated with gas. This was overcome by Mond and Langer, but only by the loss of considerable quantities of platinum from the cell. (3) Indirect cells; these include (a) oxidation-reduction cells, in which the oxidising and reducing agents present in solution in the two compartments are re-generated by fuels and air respectively; the regeneration may take place in the compartment or externally; these suffer from both current polarisation, depending on electrode area, and time polarisation, dependent on the volume of the cell; (b) metal-anode cells, in which a metal obtained by reduction with fuel is used in the solid or the liquid state as the attackable electrode. A metal of low melting-point is desirable.

The authors' experiments on semi-direct fuel cells, using nickel as the substratum of a gas-electrode, were unsuccessful. More promising results were obtained with the metal-anode type; zinc appeared unsuitable and difficulties were encountered with lead, but better results were obtained with cells (both hot and cold) in which tin formed the active element. The authors' results are, however, admittedly mainly of a negative character.

A paper by Dr. A. Ferguson described a "New Method for determining Surface Tension." Instead of measuring the height to which a column of liquid rises in a capillary tube, external pressure is applied to force the level of the liquid to the bottom of the tube, this pressure being measured by a delicate manometer. Among the advantages claimed for this method are that it avoids difficulties due to variations in tube diameter and to variations in temperature in the capillary tube itself; also the capillary tube may be very short, which facilitates cleaning. The method is rapid in use, and it gives

results which are in good agreement with those obtained by the best methods previously known.

A paper by Mr. L. F. Knapp on "The Solubility of Small Particles and the Stability of Colloids" was taken as read.

#### THE METASTABILITY OF MATTER.

Lectures on "The Metastability of Matter and its Bearings on Chemistry and Physics" were delivered on May 10 and 12 at University College, London, by Prof. Ernst Cohen, of Utrecht University, a distinguished worker in the field of allotropy and polymorphism.

The first lecture was devoted to a general description of metastability as exhibited by elements. Enantiotropy was illustrated by reference to the grey and white forms of tin, and a striking experiment was performed showing that a contraction in volume of 30 per cent. occurs when grey tin is converted into white tin, an electrolyte being added to accelerate the conversion. The explosive form of antimony was taken as an example of monotropic allotropy, i.e., of the existence of one form of an element which is unstable at all temperatures and pressures. Specimens of this form of antimony had been brought from Holland, and those which had survived the journey were shown to emit great heat when they were reconverted into ordinary antimony by scratching, 20 cal. from each gram of metal being evolved. The allotropy of non-metallic elements was discussed by reference to sulphur.

In the second lecture, Prof. Cohen reviewed the phenomena of metastability as exhibited by compounds. Polymorphism among compounds is often shown by differences in colour. Thus thallous picrate occurs in red and yellow varieties, the transition point being 36° C., but the yellow variety is obtained in a metastable form if cooled from above 36° C., and the red variety, if dry, can be heated up to 100° C. without changing into the yellow. Similar phenomena are shown by the black and red forms of mercuric sulphide. In all such cases, the conversion of the metastable form into the stable form at ordinary temperatures takes place slowly, but is accelerated by the presence of a very small amount of an electrolyte. Cadmium iodide was taken as an example of a salt existing in two forms possessing the same colour—the  $\alpha$  or stable form of sp. gr. 5.8 and the  $\beta$  or metastable form of sp. gr. 4.8. The difficulty of crystallising cadmium iodide from solutions was shown to be due to the presence of a minute quantity of cadmium hydroxide. Ordinary cadmium iodide is a mixture of the  $\alpha$  and  $\beta$  varieties. On dissolving the salt in water, the  $\beta$  form is converted to the stable  $\alpha$  form, but the crystals separated from the solution consist of a mixture of both. The  $\beta$  form can be converted completely to the  $\alpha$  form by heating to 150° C., but it has hitherto not been possible to obtain the  $\beta$  form pure; a mixture containing about 50 per cent. of the  $\beta$  variety is obtained by distilling the mixed compounds and cooling the sublimate in a stream of carbon dioxide. As further examples of metastability, reference was made to quartz, which exists in seven forms, to water, which exists in five modifications, and to calcium carbonate (calcite and aragonite). Lead nitrate exists in two modifications which exhibit a difference of about 2½ per cent. in their respective heats of solution in water. Among other substances which have been definitely ascertained to exist in more than one form are antimony iodide, potassium sulphate, barium sulphate, zinc sulphide, ammonium chloride, silver iodide, caustic soda, sodium tungstate, carbon tetrachloride, phenol, and urea. The existence of such modifications calls for a re-determination of the physical "constants" of very many substances.

## NEWS AND NOTES.

## FRANCE.

**Industrial Notes.—Chemicals and Fertilisers.**—Prices of heavy chemicals are falling gradually, especially those which a short time ago were greatly inflated owing to deficient supplies. Thus sodium sulphide, which a few months ago fetched 200 francs per 100 kg. is now selling at 65–70 fr. Recently published statistics of consumption of fertilisers show that the present supply exceeds the demand. There are large stocks of superphosphate; during the first few months of this year imports of rock phosphate from North Africa were only 165,000 t., as against 220,000 t. expected, and the price has been lowered to facilitate sales. On account of the small demand, basic slag has been re-inserted on the export free list. Ammonium nitrate is in poor demand, and owing to the arrival of 2400 t. of ammonium sulphate from Germany its price has been reduced to 30 fr. per 100 kg. But very little sodium nitrate is being purchased, the low value of the franc preventing purchases on the London market. The production of crude Alsatian potash in 1920 was 1,061,197 t., of which 450,000 t. was consumed in the country, 327,000 t. exported to America, 117,000 t. to Belgium, and 92,000 t. to England.

**Metallurgy.**—Little change is perceptible in the metallurgical market, but the acceptance of the Allied terms by Germany has induced a feeling of confidence which is benefiting the country economically and tending to make prices more stable. The high railway rates charged for metallurgical products are causing some anxiety, and there is a movement directed towards obtaining such modifications as will enable costs to be reduced. It is freely rumoured that an agreement will shortly be made between France and Italy by which:—(1) Italy will receive 100,000 metric tons of coal per month from the Saar coalfield. (2) French works will supply Italy with 35,000 t. of pig iron and 50,000 t. of semi-finished and finished products. (3) France will take from Italy 150,000 t. of iron scrap yearly, and more if Italy will purchase more pig iron from France.

**Coal.**—Owing to the coal strike in Great Britain, stocks of coal have considerably diminished during the past three weeks, particularly in the northern coalfields; they now aggregate some 600,000 t., which is still twice the amount usually held at this time of the year. Fuel consumption, however, will increase, especially when the anticipated revival in the metallurgical and glass industries takes place.

**Petroleum.**—Negotiations between France and Poland relating to the working of the Polish petroleum fields are reported to be progressing favourably. The agreement, it is stated, provides that in the event of war Poland would refuse to supply France's enemies with liquid fuel. At present French interests hold 60 per cent. of the total capital invested in the Polish oil industry and 80 per cent. of that invested in the refining industry. Many foreign undertakings are attempting to secure an interest in the industry, but Poland will only favour those that are backed by the French Government.

**The Nitrogen Industry.**—A Bill will shortly be presented to the French Chamber for sanctioning an agreement between the Ministry of War and the Société des Produits Azotés, by which the latter will take over the electrochemical works at Lannemezan and the hydro-electric works at Bordères-Louron and Lendenvielle for a period of 70 years. The company undertakes to increase the capacity of the plant to 20,000 tons of cyanamide and a similar quantity of carbide per annum, and the State will share the profits and receive certain dues.—(*Chem. Ind., Apr. 25, 1921.*)

## UNITED STATES.

**Nitrogen Fixation Plants.**—The Secretary of War has ruled that pending the further development of the nitrogen-fixation industry by private interests, the Government plants at Sheffield, Ala., and at Muscle Shoals are to be retained under Army control in the most economical stand-by condition; and the Fixed Nitrogen Research Laboratory at American University, Washington, D.C., is also to be retained and continued under the War Department.

**The Government Kelp Plant.**—The United States Government kelp-plant is to be sold as soon as possible after July 1. The plant has been operated to determine whether carbon black, iodine, and potash salts can be recovered on a commercial basis, and it has treated 100 tons of wet kelp per day from which, it is claimed, 1500 lb. of carbon and two tons of potash salts are obtainable. The Bureau of Soils is more interested in finding a customer who will continue the experiments than one willing to pay a high price. The plant is being sold in accordance with the recent Agricultural Appropriation Bill.

**Spring Meeting of the American Chemical Society.**—The meeting held at Rochester under the presidency of Prof. Edgar F. Smith during the last week in April was one of the largest of its kind; more than 1300 attended and 282 papers were presented in eleven divisions and sections. The congestion of papers has led some of the divisions and sections to appoint committees to examine and revise papers beforehand and to decide whether they shall be read in abstract or *in extenso*, or not at all.

Dr. E. K. Mees gave an address on "The Measurement of Colour," Prof. Wilder D. Bancroft on "Blue Eyes and Blue Feathers," Dr. R. E. Wilson one on "Surface Films as Plastic Solids," and Prof. Irving Muir discoursed on "The Relation between the Stability and the Structure of Molecules." The new Petroleum Section made an auspicious beginning under the chairmanship of Dr. T. G. Delbridge, of Philadelphia; among a large number of papers presented to it was one by Dr. C. F. Maberg on the separation of non-volatile hydrocarbons by fractional solubility in hot ether-alcohol mixtures.

The Division of Industrial and Engineering Chemistry held a symposium of "Drying," a paper by W. K. Lewis on "The Rate of Drying Solid Materials" being one of the more interesting. A symposium which attracted much attention was that on "Our Future Supplies of Liquid Fuel." Prof. H. N. Whitford, of the Yale University School of Forestry, urged the conversion into alcohol of vast quantities of such tropical vegetation as cassava, nipa palm, or bamboo, and two other speakers advocated utilising the enormous quantities of waste wood in the U.S. forests. Better methods of agriculture and of conserving food products were proposed in the Division of Agricultural and Food Chemistry. One speaker described new groundnut by-products; four chemists suggested improved methods for the making of ice cream; another gave hints on preventing the discoloration of canned sweet potatoes; and a most helpful paper related to improvements in the manufacture of cheese. There was also an elaborate report on means of preventing the mosaic disease of spinach.

Advanced methods of manufacture considered by the Section of Sugar Chemistry and Technology should have an important influence on the production of sugar and may tend to stabilise prices. There seemed, however, to be no indication of a drop in the cost of the so-called "rare sugars," such as mannose, which is made from the shavings of the Ecuador ivory nut discarded from the button factories, and is now \$142 per lb. These sugars are used for the detection of disease germs.

In the Dye Division a new process for producing alizarin from benzene as initial material was outlined by C. W. Schaffer, and a new method of making Malachite Green was also reported. A method for detecting and estimating carbon monoxide, based on the colour-change of a mixture of fuming sulphuric acid and iodine pentoxide, was described by C. R. Hoover; and the Division of the Chemistry of Medicinal Products discussed new non-habit-forming drugs to take the place of those narcotics which enslave. An interesting feature of the meeting was the visit to Kodak Park, the largest of the works of the Eastman Kodak Co. at Rochester, which produces about 125,000 miles of kinematograph films, uses over 1300 tons of cotton and over 150 tons of pure silver yearly.

The membership number of the Society at the end of 1920 was 15,582.

**Phosphate Rock in 1919.**—The phosphate rock marketed in the United States in 1919 amounted to 2,271,983 long tons, valued at \$11,591,268, which showed a decrease in quantity of 9 per cent. and an increase in value of 41 per cent. over the figures for 1918. The actual quantity mined was 1,851,549 tons, or nearly 19 per cent. less than in 1918. There was an increase in the output from South Carolina, Tennessee and the Western States, but production in Florida, the chief producing State, fell by 20 per cent. owing to a prolonged strike. Exports fell during the war to only about one-tenth of their pre-war volume, having declined from 1,300,000 long tons in 1913 to about 143,000 t. in 1918, but in 1919 they improved to over 378,731 t. In 1920 the total quantity marketed was 4,103,982 long tons (Florida 82%, Tennessee 16%), and the exports rose to 1,070,012 t. The market for American phosphate in Southern European States is bound to be small because France and Spain have ample supplies available from Tunis and Algeria, and have greatly increased their capacity to manufacture sulphuric acid.

*World's production of phosphate rock in 1918-19.*

	1918.	1919.
	Metric tons.	
Algeria .. .. .	198,539*	276,040
Angaur Island .. .. .	—	—
Belgium .. .. .	61,700	—
Canada .. .. .	127	22
Christmas Island .. .. .	—	—
Dutch West Indies :—		
Aruba .. .. .	—	—
Curacao .. .. .	—	10,058
Egypt .. .. .	31,147	29,364
France .. .. .	—	—
French Guiana .. .. .	—	—
Japan, including Rasa Island ..	191,722	—
Makatea Island .. .. .	—	40,000
New Caledonia, Huon Island ..	—	—
New South Wales .. .. .	305	585
New Zealand .. .. .	5,080	—
Norway .. .. .	—	—
Ocean and Nauru Islands .. ..	—	—
South Australia .. .. .	8,204	6,045
Spain .. .. .	43,303	25,035
Tunis .. .. .	821,145	—
United States .. .. .	2,530,612	2,308,448

\* Exported.

(For the world's production in 1913, 1915, and 1916, cf. J., 1920, 168 B.)—*U.S. Geol. Surv., Feb., Mar., 1921.*

**Strontium in 1919.**—Strontium ore was first mined in the United States in 1916, when, under the stimulus of high prices and war needs, 250 short tons was produced and marketed. In 1917, 4305 t. was marketed, in 1918, 400 t., and in 1919 there was no production at all. During 1919, 1393 t. of crude celestine was imported from England, and also certain manufactured salts. The production of salts, chiefly the nitrate, was 1,387,555 lb.; the carbonate was also made, mostly for use in the manufacture of other salts, but some was used for experimental work in sugar-refining. Should these

experiments lead to the general adoption of strontium compounds for the refining of beet sugar in the United States and Canada, the mining of strontium ore would be revived on a large scale. As is well known, strontium salts are used chiefly in the manufacture of fireworks, signal lights, and medicines. A little strontium is alloyed with copper in making castings, to harden the copper and to prevent the formation of blow-holes. For this purpose the alloy is obtained either by adding to the melt a little metallic strontium, or by electrolysis of fused strontium chloride, using a copper cathode. The small amount of strontium introduced into the copper does not affect its electric conductivity.—(*U.S. Geol. Surv., Dec. 5, 1920.*)

#### AUSTRALIA.

**A Proposed New Industry for Victoria.**—At Bendigo, in Victoria, works are proposed for the manufacture of paints and glazing preparations for pottery-ware. Pottery is already produced in the neighbourhood. Large deposits of red ochre and hematite in the district are to be developed, and these will provide the raw material. A site for the works has been secured close to the Bendigo-Echuca railway line.—(*Hardware and Machinery, Feb. 4, 1921.*)

**The Sugar Situation.**—It is announced that the Commonwealth Government will continue to pay Australian sugar growers £30 6s. 8d. a ton for raw sugar during the current year, wholesale and retail prices being fixed at £49 per ton (refined) and 6d. per lb., respectively. The crop for 1920-21 is expected to be heavy and will, it is hoped, suffice for requirements, in which case the price may be reduced. Up to June 30, 1920, the Commonwealth Government had lost over £900,000 on its sugar transactions, but these losses will probably be recovered by August 1, 1921. Imports of sugar during the last two years were:—1918-19, 52,569 t. (£1,052,124); 1919-20, 112,805 t. (£4,359,203); Dec. 31, 1920, 24,475 t. (£1,059,342).—(*Bd. of Trade J., May 19, 1921.*)

#### BRITISH INDIA.

**Indian Bauxite.**—In the first number of the *Journal of Industries and Labour* Dr. J. Coggin Brown, of the Geological Survey of India, contributes an article on bauxite with special reference to its occurrence and exploitation in India. The richest areas yet discovered are on the Bahir plateau, in the Balaghat district, and near Katni, in the Jubbulpore district, both in the Central Provinces. Valuable ores are also found in the Mandla and Seoni districts of the Central Provinces and in the States of Sarguja and Jashpur. The Balaghat bauxite, like that of Jubbulpore, is of very high grade, and there seems little doubt that large quantities are available; eight analyses of samples gave: Al<sub>2</sub>O<sub>3</sub>, 51.62-58.83%; Fe<sub>2</sub>O<sub>3</sub>, 2.70-10.58%; TiO<sub>2</sub>, 6.22-13.76%; and combined water, 22.76-30.72%. Occurrences of limited extent but of excellent quality exist in Western Chota Nagpur, and considerable amounts of the mineral are associated with the iron ores of the Bababudan Hills. A series of analyses of samples from the Kemman-gundi area gave: Al<sub>2</sub>O<sub>3</sub>, 34.20-62.50%; Fe<sub>2</sub>O<sub>3</sub>, 5.10-40.70%; SiO<sub>2</sub>, 0.26-13.60%; and water, 20.00-31.90%. Further deposits have been found in the Bhopal, Rewah, and Tonk States of Central India, the Satara district of Bombay, the Madras Presidency, and elsewhere. The official figures of the output of Indian bauxite, e.g., 1192 tons in 1918, in no way reflect the potentialities of the deposits, which are now being investigated by the Geological Survey with a view to publishing a memoir on the subject.

In 1907 Sir Thomas Holland stated that probably the best means of effecting the large-scale develop-



ment of Indian hauxite was to manufacture pure alumina locally by extraction with alkali for export to aluminium works or for the local manufacture of aluminium salts. Exportation of the raw or calcined material was not practicable, and the installation of aluminium works in India would involve heavy capital outlay under untried conditions and elaborate investigation before power works could be erected. Since then, however, several concessions have been taken out and more extensive knowledge of suitable sites for power stations has been acquired; but in the event of the erection of an aluminium works in India, a part of the production would have to be exported, as the Indian consumption is comparatively small. The success of aluminium enterprises in other countries has depended more on cheap electrical energy and large-scale production than on other factors.

The article also contains useful information on grades and specifications, markets, prices, and chief sources of supply.

#### JAPAN.

**Petroleum Production.**—The Department of Agriculture and Commerce, Tokyo, has issued the following statistics of production of crude petroleum and its distillation products during the period 1917–20. In the appended table the quantities are given in barrels of 35 Imperial gallons, and natural gas is included under "crude oil":—

	1917.	1918.	1919.	1920.
	Thousands of barrels.	Thousands of barrels.	Thousands of barrels.	Thousands of barrels.
Total crude oil ..	2949.4	2535.4	2350.5	2364.1
Naphtha ..	141.8	166.8	167.9	165.6
Lighting oil ..	517.8	349.4	276.8	209.9
Light oil ..	697.7	744.2	657.9	697.7
Lubricating oil ..	400.4	393.6	415.2	442.4
Heavy oil ..	622.8	376.6	224.6	209.9

—(Ch. of Comm. J., May 6, 1921.)

#### GENERAL.

**Lead Poisoning.**—Referring to Dr. Miall's remarks on lead poisoning in a recent issue of this Journal (p. 140 R), Miss Helen Masters, of King's College for Women, University of London, writes that safeguarding the health of the workers is not the only aspect of the problem, and that the possibility of the public suffering from the use of lead glazes on cooking utensils must also be taken into consideration. Miss Masters published in the *Analyst* (1919) and in the *Lancet* (1920) the results of her examination of glazed earthenware casseroles of English, French, and Dutch manufacture, which showed that practically all of them yielded soluble lead compounds on boiling with dilute solutions of organic acids such as might be present in ordinary cooking processes, and she adduced other evidence of the danger of using such lead-glazed ware. In the interests of public health she now advocates either that the use of lead glaze for cooking utensils should be prohibited, or that a standard test be adopted to which such vessels should be submitted before being placed on the market.

**Tetrachlorethane Poisoning.**—Tetrachlorethane, the non-inflammable solvent which came into prominence during the war as a constituent of aeroplane "dopes," is now used industrially as a solvent for cellulose esters, etc., in the manufacture of such articles as non-inflammable films, lacquered goods, and artificial silk. In a paper in the April issue of the *Journal of Industrial Hygiene* Dr. D. C. Parmenter, of Harvard University, describes the symptoms of tetrachlorethane poisoning and discusses methods of prevention. His observations were made in an artificial-silk factory and extended over five months, during which period twenty-one cases of poisoning occurred, but no fatalities. The initial symptoms are abnormal fatigue, discontent, general nervousness, and loss of appetite, and as the

poisoning progresses these are accompanied by nausea and vomiting. Such symptoms, although often not marked, are nevertheless detectable by careful observation, and their proper interpretation, together with a clear understanding of the importance of the blood findings, makes possible the prevention of all but the very slight cases of poisoning. Ventilation is an important preventive, and the wearing of gas-masks is recommended in special cases, e.g., in dissolving cellulose acetate and in working with acetic anhydride. Dr. Parmenter concludes that the industrial use of tetrachlorethane presents no more difficulties than that of benzol, TNT, or lead.

**Chemical Industry in Germany.**—In consequence of the "Sanctions," the demand for chemical wares has greatly diminished, and export business has practically been at a standstill since the beginning of April. It is anticipated that the exports for the first half of this year will show a very considerable falling off from those of the corresponding period of 1920, when 1,482,421 metric tons of chemicals, drugs, dyes, etc., valued at 4560 million mk., was exported (the corresponding figure for 1914 was 2,415,550 t.). The exports for Jan.-June, 1920, included:—Potash salts, 527,960 t. (807 million mk.); coal-tar dyes, 8850 t. (849 mill. mk.); indigo and indigo-carbonyl, 4506 (257 mill. mk.); alizarin and alizarin dyes, 2004 t. (134 mill. mk.). Although the nitrogen factories have been fully employed, production has seriously declined in practically all other branches of the chemical industry.

Exportation of chemicals continues under the control of the "Aussenhandelsstelle Chemie" (Export Bureau) in Berlin, which was established in June, 1919, as an autonomous organisation for the chemical industry to be conducted on strictly business lines. It is subdivided into sections corresponding to the chief divisions of chemical industry; it issues licences for importing and exporting chemical products, and fixes minimum prices for exported goods. The fees charged by the Bureau for its services are 1 mark per 1000 marks of the value for export, and 30 pf. per 1000 marks of the import value.

A small book entitled "Chemicalia" has just been published in Berlin that is designed to take the place of the well-known reference-book "Wenzel," which has not been on sale for about ten years. The new book gives the names, addresses, etc., of all the important chemical manufacturing firms, together with a list of their specialities in the German, English, French, and Spanish languages, and it contains much other information of value to producers, consumers, and traders.

**The Chemical Industries of Upper Silesia.**—Both before and during the war the chemical industries of Poland were very undeveloped, and the country's requirements in fertilisers, dyes, drugs, and substitutes for textile finishing, etc., were derived in a large measure from Upper Silesia, where chemical industries were first established in the period 1867–1880. At that time there were only about ten manufacturing firms of any consequence, and at the present time only one of these employs more than 100 full-time workers. Between 1890 and 1900 development proceeded apace, and the two largest undertakings established during this period—the Rütgers-Werke and the A.-G. Lignose Konzerns—still play an important part in the chemical industry of Upper Silesia; the former produces manufactured fuel, electrodes, etc., employs 1300 workers, and has a capital of 40–50 million marks, and the latter manufactures ammonium nitrate, nitric acid, explosives, etc., and employs over 1000 workers. The period 1900–1910 saw the establishment, in Gleiwitz, of a branch of the Griesheim Elektron company, which produces mainly oxygen, the Oberschlesische Farbwerk, Idawische (litho-

pone), the Oberschlesische Thomas-Phosphatwerke, Szcepanowitz near Oppeln (fertilisers), the Mineral-ölraffinerie Idaweihe (mineral-oil lubricants, etc.), the Oberschlesische Isoliermittel, G. C. Flegel and Co., Kattowitz, the Chemische Werke Carbon at Ratibor, and others. During the war period and after, the following new undertakings have been established:—The A.-G. Lignose, Kriewald, and the Chemische Fabrik von Heyden, Kostucha, both of which make potassium chlorate; the Oberschlesische Zellstoffwerke, Krappitz (cellulose, etc.), with a capital of 1.5 million mk.; the Oberschlesische Phosphatwerke, Oppeln, founded in 1919 with a capital of 300,000 mk. A small water-glass factory was erected in Hindenburg in 1918, and in 1920 the Oberschlesische Stickstoffwerke was established to acquire, erect, and manage nitrogen factories with a (present) capital of 250,000 mk. The Oberschlesische Zinkhütten (zinc smelters) in Kattowitz, which dates from a much earlier period, produces sulphuric acid from the roasting of blende. It will thus be seen that the chemical industry of Upper Silesia is of considerable magnitude; it comprises 60–80 undertakings, employs about 5000 workers, and commands a total capital of 150–200 million mk., exclusive of the important coke-oven plants. The coking industry has steadily developed in recent years, and early in 1920 the individual firms formed a company—the Chemische Werke Oberschlesiens—for the purpose of jointly working up and selling their entire production of tar and ammonia. The Oberschlesische Kokswerke und Chemische Fabriken, which was founded in 1890 and is now working with a capital of 35 million mk., has plants at Stalley (195 ovens), Zahrze (75 ovens), Poremba (220 ovens), and Glückauf (60 ovens); the tar is worked up by the Oberschlesische Gesellschaft für Teerverwertung, the tar oils are sold by the Verkaufsvereinigung für Teererzeugung in Essen, the pitch is taken by the Upper Silesian briquette factories, and the ammonia is converted into sulphate and distributed through the Nitrogen Syndicate in Berlin.—(*Chem.-Z.*, Apr. 28, 1921.)

**Austrian Magnesite.**—Owing to the extraordinary demand for this mineral, many developments are taking place; new and valuable deposits have been discovered and exploited, and production is to be carried on at maximum capacity. The mines are all situated in present-day Austria, and all the more important belong to the Veitsch and Trieben mines in Styria and the Breitenau mine in Lower Austria. The life of the Veitsch mines is estimated to be 200 years at the present rate of production. Last year a French-Swiss group, which included the Schneider-Creusot Co., acquired an interest in this undertaking; profits were 23 million kronen and a dividend of 40 per cent. was paid (4 mill. kr. and 10 per cent. in 1919). Among recent developments may be noted the formation of the Steirische Magnesitindustrie A.-G., with plants at Oberdorf and Neuberg, the discovery of promising deposits at Mayrhofer, acquired by the Veitsch company, and the working of valuable deposits near Kraubath; the Salzburger Magnesit Industrie Gesellschaft is planning the exploitation of mines in the Salzbnrg country, and a new company is operating the mines at St. Martin in Styria. During 1913, 123,900 t. of the calcined mineral was exported, and in 1920 about 63,000 t.—(*Times Tr. Supp.*, May 14, 1921.)

**German Beet-Sugar Production in 1920–21.**—The German beet-sugar crop for 1920–21 is estimated to be about 1,200,000 tons, an amount which will enable household rations to be increased and permit of distribution to all industries. It is announced that the internal tax on sugar is to be increased to 100 mk. per 100 kg., and with the mark at its present value the result would be to favour export

of sugar, as in comparison with the world value the tax is nominal on the prices made by exported sugar. Germany's consumption, however, is much higher than the production, and export is unlikely.—(*Ind. Tr. J.*, Apr. 1, 1921.)

**Production of Blackstrap Molasses in Cuba.**—The estimated production of blackstrap molasses in Cuba in 1920 was 180 million gallons (U.S.A.), of which 20–30 million galls. was used locally in the production of fuel alcohol. The storage and conservation of the molasses by the sugar mills largely depends on the market price, and much of it is burned at the sugar factories as fuel. The demand is expected to increase, especially if alcohol comes into use as a motor fuel. Shipments of blackstrap molasses from Cuba amounted to 199,193,722 galls. in 1917, 163,716,219 galls. in 1918, and 49,782,231 galls. in the first six months of 1919.—(*U.S. Com. Rep.*, Mar. 5, 1921.)

**Sugar-Beet Cultivation in Shantung.**—The announcement that a beet-sugar factory is to be established at Wangtaichiao, near Tsinanfu, by a Chinese company, the Pu Yih Industrial Co., marks the first attempt to establish a beet-sugar industry in Shantung. Machinery is being obtained from the United States and Japan, and sugar-beet seed is being distributed in the districts around Tsinanfu. Favourable conditions of soil, climate, and water supply appear to obtain in Shantung, and with abundant cheap labour, the success of the enterprise would seem to be assured. The company has secured a monopoly of the industry for 24 of the 100 or more districts in Shantung.—(*U.S. Com. Rep.*, Mar. 5, 1921.)

**The World's Petroleum Output in 1920.**—The American Petroleum Institute estimates the world's production of petroleum in 1920 at 688,474,251 barrels, which represents an increase of 24.2 per cent. over the previous year. The estimated production in barrels by countries is as follows:—

	1919 Barrels.	1920 Barrels.
United States .. .. .	377,719,000	443,402,360
Mexico .. .. .	87,072,954	159,860,000
Russia .. .. .	34,284,000	39,000,000
Dutch East India .. ..	15,789,000	16,000,000
India .. .. .	8,453,000	8,500,000
Rumania .. .. .	6,517,748	7,468,818
Persia .. .. .	6,289,812	6,604,734
Galicia .. .. .	6,255,000	6,000,000
Peru .. .. .	2,561,000	2,700,000
Japan and Formosa .. ..	2,120,500	2,213,639
Trinidad .. .. .	2,780,000	1,628,637
Argentina .. .. .	1,604,300	1,566,926
Egypt .. .. .	1,602,184	1,389,213
France (Alsace-Lorraine) ..	—	700,000
Venezuela .. .. .	321,396	500,000
Canada .. .. .	220,100	220,000
Germany .. .. .	925,000	215,340
Italy .. .. .	38,254	38,000
Total .. .. .	554,505,048	688,474,251

In 1920 the United States produced 64.4 per cent. and Mexico 23.2 per cent. of the world's output.—(*Min. Mag.*, Apr., 1921.)

**Wood-pulp Manufacture in Alaska.**—The United States Department of Agriculture has recently received samples of the first run of a new pulp-mill established at Juneau, in the Tongass National Forest. The mill, the first to be established in the territory, is operated by hydro-electric power, and has a capacity of 20 tons a day, with power resources available for increasing the output to 250 tons. So far spruce has been utilised, but hemlock will be ground for newsprint purposes, and the eventual manufacture of paper at the mill is planned. An area of the Tongass National Forest containing 2000 million feet of pulp-wood will, it is stated, shortly be placed on the market in response to inquiries from prospective paper manufacturers.—(*Id.* of *Trade J.*, Apr. 14, 1921.)

**Saccharin Monopoly in Yugoslavia.**—The Government monopoly of saccharin and other sugar substitutes in Yugoslavia has hitherto been effective in Bosnia and Herzegovina only, but it is now to be applied to the whole State.—(*U.S. Com. Rep.*, Apr. 1, 1921.)

**Peppermint Distillation in Piedmont.**—The cultivation and distillation of peppermint in Piedmont is a small but very profitable industry, as the demand greatly exceeds the production. In 1920 the crop from 1483 acres was 22 million lb., which on distillation yielded 59,525 lb. of peppermint oil, valued at 6 million lire (£240,000 at par). It is anticipated that the crop for 1921 will be roughly 20 per cent. larger than that for 1920, as the distillers have promised higher prices to cultivators in order to increase production.—(*U.S. Com. Rep.*, Apr. 13, 1921.)

**Fuel Oil from Sicilian Asphalt.**—One of the richest areas of the asphalt deposits in Sicily is owned by an Italian company which, in addition to working up the material into products for paving and roofing, has developed a process for the production of mineral oil with a very low bituminous content and a calorific value of 10,300 calories per kg. A large battery of gas furnaces is employed, and the heat of the ash is almost completely recovered by means of a current of the inert gases from the condenser discharges; particles of oil carried over in the combustion fumes are recovered by means of centrifugal filters and condensers. The output for 1920 is estimated to be 5000 metric tons of oil, and is hoped to increase this to 10,000 t. in 1921. The company is planning the erection of a large refinery near Rome.—(*U.S. Com. Rep.*, Feb. 11, 1921.)

**Sumac Production in Sicily.**—Sicily is the largest producer of sumac in the world, the United States taking second place. The annual output in recent years is estimated at 15,000 metric tons, which is 50 per cent. below the pre-war production. This decrease is due to diminished cultivation, which is believed to be only temporary. Male sumac is found in the provinces of Palermo and Girgenti, and contains 28 per cent. or more of tannin, whereas female sumac, found mainly in the provinces of Catania and Girgenti, contains an average of 24 per cent. There are about eight large sumac-grinding mills in Palermo, but no factories for preparing the extract, although it is considered probable that they will be built when the trade revives. Exportation from Palermo is chiefly directed to Great Britain, the United States, France, and Germany (*cf. J.*, 1920, 263 n).—(*U.S. Com. Rep.*, Mar. 23, 1921.)

**Columbian "Pita" Fibre.**—The term "pita" is used very widely in Central and South America, and is applied to several fibre plants (including sisal hemp) and also to string. Colombian "pita" belongs to the *Bromeliaceæ* and is a species of *Ananas*, closely allied to the pineapple; it grows in the shade of forest trees, forming extensive, impenetrable areas known as "pitales," but it can be grown on light open land and at altitudes ranging from sea-level to 3000 ft. The fibre is said to be superior to sisal in strength, fineness, and durability, and if available commercially it would compete with sisal hemp in the manufacture of rope and twine. The average annual yield of the forest "pitales" is estimated to be 45.5 tons of leaves containing 5 per cent. of fibre. By the native method of extraction about 40 per cent. of the fibre is wasted, so that if 3 per cent. could be obtained by mechanical extraction, the yield would be about 1.5 tons of dry fibre per acre. Certain advantages are claimed from the harvesting of fibre from the natural "pitales" as compared with cultivation of sisal plantations, but the difficulty is that no machine is yet available for extracting the fibre from the leaves.—(*Imp. Inst. Bull.*, XVIII., No. 4.)

**The Industrial Value of Perilla Seed.**—In view of the increased demand for drying oils, it has been thought that the cultivation of Perilla seed (*Perilla ocymoides*) might be encouraged in British possessions; at present it is grown for commercial purposes principally in Manchuria, China, and Japan. To this end the Imperial Institute sent out samples of Japanese seed to India, East Africa, South Africa, Rhodesia, and Cyprus for trial cultivation, and a report from Cyprus is now published in the *Bulletin of the Imperial Institute*, Vol. XVIII, No. 4. The seeds contained 6.3 per cent. of moisture and yielded 43.1 per cent. of oil (46 per cent. on a dry basis) which possessed the properties of Perilla-seed oil, and which should be readily saleable in this country for paint and varnish making and other purposes for which a drying oil is required. The residual meal contained 35.6 per cent. of protein and 18.9 per cent. of fibre, and would probably be a valuable feeding stuff; trials, however, are required to determine this point. The authorities in Cyprus have been asked to continue the experiments.

**Finsbury Technical College.**—The threatened closure of this valuable institution having been averted (*cf. J.*, 1921, 150 n), the Governing Body announces that the entrance examination for the session 1921–22 will be held on September 20 next. Applications for admission should be forwarded to the College not later than September 15 on forms to be obtained from the Registrar, Leonard Street, City Road, E.C. The programme of the College is under revision and will be issued in due course.

## PERSONALIA.

Mr. W. B. Worthington has been elected president of the Institution of Civil Engineers for the year 1921–22.

The list of newly elected foreign members of the Royal Society includes the names of Prof. Albin Haller and Prof. P. Zeeman.

Mr. C. R. Delong has succeeded Dr. Grinnell Jones as chief of the Chemical Division of the United States Tariff Commission.

Senator Piero Ginori Conti is succeeding Prof. G. Ciamician as president of the Associazione Italiana di Chimica Generale ed Applicata.

The Willard Gibbs Medal of the Chicago Section of the American Chemical Society is to be presented to Madame Curie at a special meeting to be held on June 14.

The University of Glasgow is to confer the honorary degree of LL.D. upon Sir Dugald Clerk and Prof. J. C. Irvine, principal of St. Andrew's University.

Prof. P. H. M.-P. Brinton, head of the chemical department in the University of Arizona, has been appointed professor of analytical chemistry in the University of Minnesota.

The Court of the University of Manchester has conferred the honorary degree of Doctor of Science upon Sir E. Rutherford, formerly professor of physics in the University.

Sir J. J. Thomson has been elected honorary professor of natural philosophy, and Sir Ernest Rutherford professor of natural philosophy, in the Royal Institution of Great Britain.

The death of Dr. E. B. Ross, chief physicist to the Bureau of Standards, Washington, D.C., is announced by *The Times*.

## PARLIAMENTARY NEWS.

### HOUSE OF COMMONS.

#### Wage-Reduction in Industries.

In a long written reply to Sir W. de Frece Dr. Macnamara gave a list of the industries in which wages have been reduced in recent months. The list includes chemicals; soap and candles; bleaching, dyeing and finishing; iron-ore, shale, fireclay, and gypsum mining; limestone quarrying; iron and steel; coke and by-products; tinplate; glass-bottle manufacture.—(May 6.)

#### Arseno-Benzol Treatment of Venereal Disease.

Replying to Mr. R. Richardson, the Minister of Health (Sir A. Mond) stated that 22 deaths had been reported from the administration of arseno-benzol at centres for the treatment of venereal disease; in three of these cases death was not due to the drug, and in five others it was not certain how far the drug had contributed to the fatal result. In the last complete year for which statistics were available the administration of the drug might have been a contributory cause of, roughly, one death in every 1300 patients treated for syphilis, and of one death in every 30,000 injections. The suggestion that such information as the above should be included in a Departmental report would be considered.—(May 12.)

#### Home-Grown Sugar, Ltd.

Mr. Young, in answer to Lieut.-Col. Nall, said that it was proposed to make an advance, under certain conditions, to Home Grown Sugar, Ltd., on second mortgage of the sum of £125,000 recently voted for the purpose on the estimate of the Ministry of Agriculture. In addition, Parliament would be asked, if necessary, to provide in each of the ten years ending March 31, 1930, a sum that with the divisible profits of the company would enable 5 per cent. per annum to be paid on the £250,000 ordinary shares issued to the public, such advances to be repayable out of future profits. It was hoped that the arrangements would not prove to involve any subsidy, but in any case the agreements made between the Government and the company could not be reconsidered (*cf.* J., 1920, 223 n, 1921, 108 n).—(May 13.)

#### German Reparation (Recovery) Act.

Mr. Chamberlain, answering Capt. Wedgwood Benn, said that the Government would not repeal or suspend the above Act until it had had further experience of the working of the terms to which Germany had just subscribed, but the rate of duty would be reduced to 26 per cent., and its collection here would be in lieu of and not in addition to the general levy on all exports collected in Germany.—(May 13.)

#### Safeguarding of Industries.

The two financial Resolutions dealing with "key" industries, dumping, and depreciated exchange (*cf.* J., 1921, 131 n) were considered on May 9, 10, and 11, and passed without amendment. In introducing the first Resolution (relating to the 33½ per cent. *ad val.* duty on certain imported articles) the President of the Board of Trade based his argument largely on the needs of national defence, and stated (*inter alia*) that the prospects of the chemical industry were illimitable and its development absolutely necessary. Mr. Jameson (West Edinburgh) spoke of the grave danger to which the rubber-tyre industry was exposed owing to competition not only from the United States, but from France and Italy, and of the present widespread unemployment in that industry. Sir J. Norton-Griffiths

urged the claims of the gas-mantle industry to be safeguarded, stating that at the present time the works at Wandsworth were virtually shut down. Mr. Hannon asked for protection for the manufacture of electrodes and vitrified grinding wheels. Dr. Murray opposed the inclusion of optical glass on the ground that microscopes, etc. would become dearer to students of science. Mr. Kiley, in moving to omit beakers, flasks, etc., said that "lamp-blown ware" included common lamp-shades, and a beaker was practically "a glass jug without a handle"; he could not see how the latter could help to win a war. Mr. Lyle-Samuel, in alleging the inferiority of British chemical glassware, said that it appeared to "undergo some form of chemical dissociation" in use, and that it had a "shorter life when exposed to atmospheric conditions." All the proposed amendments were defeated by large majorities.

## OFFICIAL TRADE INTELLIGENCE.

(From the Board of Trade Journal for May 12 & 19.)

### OPENINGS FOR BRITISH TRADE.

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W. 1, from firms, agents or individuals who desire to represent U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number.

Locality of firm or agent.	Materials.	Reference number.
British Honduras..	Matches, drugs, medicines, soap, rope	604
British West Indies	Matches, paint, paper, galvanised iron	624
Canada .. ..	Pottery, aluminium ware	600
New Zealand ..	Glassware	602
South Africa ..	Mining and farmers' supplies	623
Bulgaria .. ..	Sheet iron, galvanised iron, tinplate, blackplates	605
Denmark .. ..	Laboratory supplies	629
France .. ..	Writing and printing paper	608
" .. ..	Pharmaceutical products, crude drugs, dyes	609
Belgium .. ..	Bar iron, crucible steels	610
Serb-Croat-Slovene State	Drugs	615A
Switzerland ..	Oleake	632
United States ..	Chemicals, dyes	618
Argentina .. ..	Heavy and fine chemicals	619

**MARKETS SOUGHT.**—A Canadian manufacturer of bleached sulphite wood pulp invites inquiries from U.K. paper makers. [Inquiries to the Canadian Government Trade Commissioner, 73, Basinghall Street, London, E.C. 2.]

A merchant in British Honduras wishes to get into touch with U.K. importers of logwood, fustic, chicie, and rubber. [604.]

### TARIFF. CUSTOMS. EXCISE.

**Brazil.**—The sanitary stamp tax on pharmaceutical specialities has been revised.

**British Guiana.**—The forms of certificates of origin for goods imported under the British Preferential tariff are given in the issue for May 12.

**Canada.**—Hard soap (not being whale-oil or castile soap) is rated for duty under Tariff item No. 228 when the fair market value thereof at the place of manufacture abroad is over 9 cents per lb. wholesale.

*Denmark.*—All goods arriving at the free port of Copenhagen are now free of export duty.

*Egypt.*—Benzine and mineral lubricating oil pay customs duty at the rate of 15 per cent. *ad valorem*.

*Federated Malay States.*—An export duty of 2½ per cent. *ad valorem* has been levied, as from January 1, on china clay, potash and soda felspar, and china stone.

*France.*—The export of glucose and of certain kinds of rags is now permitted.

*Germany.*—Among the articles that may be imported without licence, as from April 29, are cork, tanning barks, certain raw hides and skins, raw bones, iron ore, manganese ore, ozokerite, and montan wax.

As from April 29 export licences are no longer required for cork, certain tanning barks, rubber, gutta percha, balata, ozokerite, montan wax, sulphite lyes, soft soap, turkey-red oil, "Creolin," glycerin, paraffin ointment, cart grease, copper sulphate, cryolite, artificial resins, tanning extracts, glue, asbestos manufactures, wire rope, aluminium powder and foil, lead bars, sheet zinc, etc.

*Gold Coast.*—The import duties on spirits have been revised with effect from April 27.

*Japan.*—The proposed changes in the import tariff affect, *inter alia*, olein, stearin, paraffin wax, caustic potash and soda, sodium peroxide, potassium, cocaine, cocaine hydrochloride, spiegleisen, ferromanganese, certain kinds of iron, steel and zinc.

*Kenya.*—The import of spirits is prohibited unless they have been matured by storage in wood for at least 3 years.

*Netherlands.*—It is proposed to increase the import duties on beer, candles, chocolate, cork, gunpowder, oil (not specially mentioned), platinum, refined salt, soap, sugar, dextrose, caramel, wood spirit, and alcohol.

*Nigeria.*—Among the articles of which the import by post is prohibited are German dyestuffs and spirituous liquors.

*Poland.*—The customs duties have been suspended until July 15 on condensed milk, fertilisers, train oil, margarine, graphite, antimony, sodium sulphate, sulphuric, nitro and hydrochloric acids, dye earths, tanning extracts, pig iron, copper ingots, lead ingots, wood pulp, printing paper, and manilla cord.

Reduced rates of import duty are payable on olein, leather for the textile industry, asbestos sheet and yarn, certain wares of glass and porcelain, hydrosulphite and sulphide of soda, nitrates of thorium, cerium, beryllium, aluminium and magnesium, naphthols and sulphonic derivatives, and sulphuric ether.

*Spain.*—The export of 20,000 metric tons of olive oil is allowed under licence subject to export duty.

The customs duties on cement have been reimposed as from May 3.

*Switzerland.*—The import of certain wares of iron, copper and copper alloys is prohibited except under licence as from May 3.

All goods may now be exported under general licence with exceptions which include condensed milk, iron filings or turnings, scrap iron, indigo and coal-tar colours not specially mentioned in the general tariff.

*Trinidad and Tobago.*—The complete schedules of customs duties are set out in the issue for May 12.

Goods that are free of import duty under the British preferential tariff include blasting gunpowder, butter substitutes, some cattle foods, raw cocoa, coconuts, certain kinds of glassware, raw rubber, balata, chicle, raw hides and skins, lard, lard substitutes, lime, manure, condensed milk with not less than 7 per cent. of fat, copra, edible oils, scientific apparatus, certain tanning and dyeing materials, tonca beans, serums, and radium.

## COMPANY NEWS.

### BRITISH COTTON AND WOOL DYERS' ASSOCIATION, LTD.

The twenty-first annual meeting of the Association was held in Manchester on May 13. Mr. A. Hoegger, who presided, said that few would care to experience a repetition of the abnormal conditions which had prevailed during the last six months, or again share in losses similar to those sustained by the textile industries in consequence of the great and sudden fall in prices. Since November last few of the branches of the Association had been able to find work for their employees for more than two or three days a week, but relations with labour continued very cordial; the standard rate of wages for the 48-hour week rose to 80s. 2d. in February last, but had recently been reduced to 70s. 6d.; pieceworkers could earn far higher wages. A comparison of costs for the years ended March, 1914 and 1921, showed that the cost of dyewares had increased by 270 per cent., that of coal by 217 per cent., and that of wages by 196 per cent. Recently there had been a revival of inquiries for all classes of work dealt with by the Association, and, given more settled labour conditions, the textile trades should once again enter upon a period of prosperity.

Referring to the dyestuff situation, Mr. Hoegger quoted the official report which stated that the value of imported coal-tar colours from Jan.—June, 1920, exceeded £5,000,000. Germany, he said, was now making dyes at the rate of about 5000 tons per month, and it had been estimated that the world's output capacity was at least double that of the normal consumption. Excellent work was being done by British makers, but we were not yet entirely independent of Germany; during the past year many good and useful colours not previously made in this country were marketed, but the prices of the vat and other fast dyes were still too high to allow of an extensive business being done. Great progress was necessary to enable our textile trades to meet the ever-increasing competition in dyed and coloured woven goods and prints containing the brilliant and fast dyes so much admired and in such great demand all over the world.

### MAYPOLE DAIRY CO., LTD.

Sir W. George Watson, addressing the annual meeting held in London on May 18, said that the increased cost of labour and shorter working hours had greatly augmented manufacturing and distributing expenses, and that it would be impossible to maintain present wages and hours unless output and efficiency could be increased or more mechanical work adopted. Already the directors had considered the advisability of closing down certain departments in the factories, as foreign manufactured oil, margarine, etc., could be bought at lower prices than they could be manufactured in this country. The best "Maypole" margarine was now being retailed at 8d. per lb., compared with 1s. 1d. in December last. There had been a large increase in turnover during the past year, but the profit was less than 3 per cent. on the value of the goods sold—£36,500,000. The difficulties of trading when market values were constantly changing was illustrated by the necessity of having to purchase and carry at least 30,000 tons of crude oil three to six months before it could be delivered. During last year the price of crude oil fell from £100 to £70 per ton and the decrease in value would have seriously affected last year's results had it not been possible to reclaim some of the excess profits duty paid in recent years.

**FANTI CONSOLIDATED MINES, LTD.**—In his address at the annual meeting held on May 12, Mr. Edmund Davis, chairman, said that it had been decided to expend £250,000 in developing and equipping the company's manganese proposition in West Africa, and that a contract had been entered into for the sale of 500,000 tons of ore at a guaranteed minimum profit of 7s. 6d. per ton. Although no washing-plant had yet been erected, 85·4 per cent. of the output had averaged 49 per cent. or over of manganese. Development work during the past year indicated that previous estimates of the extent of the ore deposits were likely to be exceeded. In his opinion the manganese deposits, which were valued in the company's books at £1000, were worth the entire capital of the company (£540,000).

**NEW CAPITAL ISSUES.**—*Brunner, Mond and Co., Ltd.*—The issue at par of 2½ million 7½ per cent. cumulative preference shares of £1 each was quickly oversubscribed and the shares quoted at a premium. The money provided by this issue will be used to repay advances towards the construction of large works now being erected for the manufacture of soda ash at Wallerscote, near Warrington, for the nitrogen works at Billingham, Stockton-on-Tees, for workmen's houses, and for further working capital.

*The United Glass Bottle Manufacturers, Ltd.*, has offered £600,000 seven per cent. first mortgage debenture stock (free of income tax up to 6s. in the £) at par; the proceeds of the issue will be spent on constructional work at the Charlton factory, etc.

## LEGAL INTELLIGENCE.

### CUSTOMS SEIZURE OF PYROGALLIC ACID. *Attorney-General v. Brown.*

In the Court of Appeal on May 10, Lords Justices Bankes, Warrington and Scrutton by consent allowed an appeal (upon terms not stated) by the Crown against a judgment of Mr. Justice Sankey delivered on December 7, 1919 (*cf. J.*, 1919, 481 n.). The suit was by the Crown, by information, for the condemnation of six packages of pyrogalllic acid consigned to and imported into Manchester by the defendant, Mr. John Brown, trading as Brown and Forth, chemical manufacturers, of Manchester and London. The goods were seized on August 29, 1919, under a Proclamation dated June 25, 1919. Mr. Justice Sankey held that the Proclamation was illegal and invalid and entered judgment for the defendant with costs.

The Attorney-General now stated that the case was met by Section 4 of the Indemnity Act, 1920, and counsel for the defendant intimated that he could not contest that proposition.

### DISPUTE OVER ZINC-OXIDE RECOVERY PLANT. *Keeling and Walker, Ltd. v. The Sturtevant Engineering Co., Ltd.*

In the Court of Appeal on May 9 and 10, before Lords Justices Bankes, Scrutton, and Warrington, Messrs. Keeling and Walker, Ltd., chemical manufacturers, appealed against an order of the King's Bench Divisional Court and a judgment of the Official Referee disallowing their claims against Messrs. The Sturtevant Engineering Co., Ltd., of London, for damages for the alleged failure of a zinc-oxide recovery plant supplied by them (*cf. J.*, 1920, 403 n.).

Counsel for the appellants stated that the respondents claimed that the contract was merely for the sale of goods, but his clients contended that it was also for design and labour, and these could not be taken back. For the respondents it was admitted that they had to return the purchase price, but as the appellants had cut up the cooling-plant there should be some deduction on that account.

In giving judgment, Lord Justice Bankes held that the Official Referee was right in taking the view that the respondent company was entitled to succeed. The Divisional Court agreed with that view, but referred the matter back to the Official Referee, as there was more than one contract and different considerations might apply to the cooling plant. The respondent company had taken back the suction plant and was therefore under no obligation to pay damages, but it must return the purchase price. The judgment of the Official Referee would stand in respect of the suction plant, but if the parties could not come to an agreement as regards the cooling plant, the matter must go to the Official Referee.

## REPORTS.

**REPORT ON THE ECONOMIC, FINANCIAL, AND INDUSTRIAL CONDITIONS OF FINLAND IN 1920.** By C. H. MACKIE, *H.M. Consul, Helsingfors. Department of Overseas Trade. Pp. 44. London: H.M. Stationery Office. 1921. Price 1s. net.*

Finland was declared an independent sovereign State in December, 1917, and a Republic in July, 1919, and it was only after the latter event that the country began to recover economically. The progress made has been marvellous. With a total population of only 3,331,814 (January, 1919), mostly engaged in agriculture, Finland is one of the most prosperous of the smaller European States, a position which it owes mainly to the timber and pulp and paper industries (*cf. J.*, 1921, 71 n.). The latter made good progress in 1920 (figures of production for the whole year are not yet available), but the trade in timber was chequered. All business was adversely affected by the fluctuating rate of exchange; the official rate in London varied between 65·70 Finnish marks to the £ in March to 180 marks at the end of November, the average rate being about 102 marks (par value 22·25 mk.). Rapid progress is reported in the leather industry, which now comprises 26 tanneries and eight shoe factories. In spite of restrictions on imports and depreciated exchange, hides were imported to the value of 70 million Finnish marks. Among new developments to be noted are the erection by the Government of a sulphuric acid and superphosphate factory at Kotka, with a capacity of 20,000 t. of superphosphate per annum; the rebuilding of the Kotka cellulose factory for the manufacture of easy-bleaching sulphate pulp; a paper mill with an output of 12,000 t. of strong wrapping paper has been completed at Björneborg, and at the same place a sulphite-pulp mill capable of producing 14,000 t. of pulp per annum is nearing completion. A large hydro-electric station is being built at Aetsa to supply about 7000 h.p. to the industrial establishments at Björneborg, some 70 km. distant.

The import and export statistics given in the report cover the first nine months of the year. During this period Germany supplied the bulk of the dyes and colours and had rather more of the trade in drugs and chemicals than had Great Britain, but the United States had the greatest share of this trade. Great Britain was the largest



supplier of gums, asphalt, resin, rubber, hides, minerals and manufactures thereof, perfumery, textiles, thread, and twine. Sweden, Germany, and the United States are our chief competitors, Sweden supplying mainly metals, enamelware, and machinery, and the United States chiefly oils and fats, machinery, gums, asphalt, etc., and hides.

A summary of the trade returns for the whole of 1920 is given in the *Board of Trade Journal* for March 24. These show that although imports still exceeded exports the position in this respect has very much improved. The following are the values in millions of Finnish marks:—

	Imports. 1920.	Exports. 1920.
Total for the year .. ..	2620.4	2906.6
Chemicals and drugs .. ..	109.5	1.2
Colours, etc. .. ..	43.9	0.2
Explosives .. ..	9.3	19.4
Fertilisers .. ..	21.4	—
Ether, alcohol, etc. .. ..	5.3	2.6
Oils, fats and waxes .. ..	197.0	1.5
Asphalt, tar, resin, rubber, etc. ..	81.7	6.5
Minerals and their products .. ..	156.5	5.0
Metals and manufactures .. ..	529.8	4.8
Skins, furs, leather .. ..	121.9	38.1
Paper pulp, cardboard, etc. .. ..	6.3	1030.7
Wood and wooden goods .. ..	16.0	1633.4

Further statistics supplied by H.M. Consul show that Great Britain headed the list in the import trade with about 28 per cent., the United States was second with about 21 per cent., followed by Germany with 17, and Sweden with 10 per cent. respectively. The United Kingdom's share of the Finnish export trade was 43 per cent.

#### THIRTY-EIGHTH REPORT OF THE COMPTROLLER-GENERAL OF PATENTS, DESIGNS, AND TRADE MARKS, 1920. London: H.M. Stationery Office. 1921. Price 2d. net.

The volume of business done by the Patent Office during 1920 again showed a substantial increase, though not to the same extent as in 1919 (*cf. J., 1920, 292 B*), the total number of patent applications filed being 36,672, or 3819 more than in 1919, when the increase amounted to 11,014. Although more patent applications were filed than in any other year, a considerable proportion of the increase was due to the many foreign applications made under the Peace Treaties. Thus, Convention applications made under the extensions of time granted by the Peace Treaties and the Berne Agreement of June 30, 1920, were largely responsible for the increase of 49 per cent. in the number of complete specifications filed on application; the number of complete specifications filed after provisional specifications decreased slightly. Designs applications numbered only 13,669, or 425 less than in 1919, but trade-mark applications, with a total of 14,064, showed a gain of 1585, and were well above the pre-war level. There was a further substantial increase in the business transacted: 263,000 letters and correspondence forms and 92,000 parcels of publications were dispatched, and 260,368 patent specifications sold.

The number of hearings upon oppositions to the grant of patents was 43, and there were seven appeals to the Law Officer; 1550 hearings were given under Sections 7 and 8 of the Acts, with three appeals to the Law Officer.

Altogether 120,512 readers made use of the Library, the increase of 21,894 possibly being due to some extent to the extension of the hours to 9 p.m. The Library, which was being re-classified, contained 59,394 works (183,675 volumes, exclusive of duplicates).

The sum of £420,472 was received for patents fees (renewals £277,500), and £8690 and £39,295, respectively, for designs and trade-marks fees. Total receipts amounted to £487,543 8s. 9d. and total expenditure to £401,036, the balance representing about 18 per cent. of the total receipts.

## TRADE NOTES.

### BRITISH.

**The Fertiliser Trade in South Africa.**—The use of fertilisers in South Africa was steadily increasing until conditions arising out of the war greatly reduced importation. The decline in importation is illustrated in the appended table, which gives the imports in 1913 and 1919, and the percentages supplied by the chief countries of origin:—

	1913.		1919.	
	Long tons.	Per cent.	Long tons.	Per cent.
Basic slag ..	5,330	Ger. 43, Belg. 22, Holl. 21, U.K. 14	1,714	Holl. 100
Bone fertilisers ..	4,209	Ger. 27, Ind. 22, U.K. 21, Holl. 14, Russ. 12	112	Arg. 87, Ind. 8, U.K. 2
Guano ..	313	Seychelles 73, U.K. 15	1,778	S.W. Protect. 76, Falk. Is. 23
Nitrate of soda ..	65	Ger. 61, Chile 31	—	—
Phosphate, raw ..	680	Ger. 68, U.K. 20, Belg. 8	5,852	Egypt 96, S.W. Protect. 4
Potash fertilisers ..	1,739	Ger. 78, U.K. 7, Ind. 8	406 (lb.)	U.K. 100
Sulphate of ammonia ..	358	U.K. 92, Ger. 8	—	—
Superphosphates ..	36,619	Holl. 66, Ger. 19, U.K. 7	740	U.K. 100
Whale manures ..	—	—	9,056	Falk. Is. 100
Other fertilisers ..	7,665	U.K. 51, Holl. 35, Ger. 11	13	Nor. 45, Swed. 41, U.K. 14

The fertilising constituents required by South African soils are, in order of importance, phosphoric acid, lime, nitrogen, potash.—(*U.S. Com. Rep., Mar. 18, 1921.*)

**Sierra Leone in 1919.**—Although the trade of this Colony was satisfactory in 1919 and showed signs of expansion, there was much distress among the inhabitants owing to the shortage of food and the increased prices of imported goods. Towards the end of the year conditions improved owing to better prices being obtained for palm kernels and palm oil, which were produced and exported in much larger quantities than in former years. The crop of kola nuts was also larger, and the higher prices obtainable in Europe for piassava caused this commodity to be shipped in larger quantities, although the actual export is almost insignificant in comparison with the vast amount of raw material available. The gum copal reserve in the Kessewe Hills and four areas in the Ronietta district were opened to gum collectors during the year. So far as is known the mineral resources of Sierra Leone are very small; there are, however, deposits of laterite iron ores which would be valuable if a process for treating titaniferous iron ores were available. Various products of the Colony were examined by the Imperial Institute. Po-yak nuts (*Parinarium* sp.) were found to yield a drying oil similar in value to tung oil, and a meal with a lower protein content than is usual in oilseed residues; both nuts and oil should be readily saleable in the United Kingdom. Rowe fibre was also examined, and reported to be very coarse, harsh, interlaced, and only suitable for making heavy-sized yarns or coarse string and rope. Imports were valued at £2,034,780 and exports at £2,101,569, both showing large increases over the preceding year. The chief exports were:—Palm kernels, 50,622 tons; palm oil, 823,750 galls.; kola nuts, 2995 t.; ginger, 1069 t.; and piassava, 1059 t.—(*Col. Rep.-Ann., No. 1065, Mar., 1921.*)

### FOREIGN.

**The Dutch Oleomargarine Trade in 1920.**—Despite the general trade depression and wide variations in prices, the exports of margarine from Holland

during 1920 showed a notable increase, much of which was due to the development of new markets. The anticipated increase in trade with Germany did not materialise, but Great Britain took very much more of the output than formerly. The exports of oleomargarine and butter substitutes in 1919 and 1920 were 53,881 and 92,883 metric tons, respectively, the percentages taken by the principal consumers during the two years being as follows:—United Kingdom, 48 and 55; Germany, 46 and 33; Belgium, 4 and 2; Czechoslovakia, 5 (1920 only). The raw materials are obtained mainly from the United States or through firms in other countries which are controlled by American capital.—(*U.S. Com. Rep.*, Mar. 24, 1921.)

## REVIEWS.

LEHRBUCH DER FARBENCHEMIE. By DR. HANS TH. BUCHERER. Second edition, revised. Pp. xii. + 636. (Leipzig: Otto Spamer. 1921.) Prices: unbound 47s. 1d., bound 51s.

It is a far cry from Dr. Bucherer's modest little booklet of 1904 in the "Sammlung Götschen" to the present imposing volume, but it indicates that the lines originally laid down by the author have proved to be sound under the test of experience. The second edition of Prof. Bucherer's larger work differs but little from the original 1914 edition except that it is slightly longer (636 as against 557 pages), whilst the text has been revised and some new matter added, but all the excellent features which distinguished the former edition are retained, so that the text-book still remains one of the best of its kind for students of colour technology. The section dealing with the anthocyanins is entirely new, but in spite of the large amount of work done by English chemists on this class of colouring matters, almost the only name mentioned is that of Willstätter, and the same tendency to ignore the work of non-Teutonic chemists is noticeable in the "Literatur Verzeichnis" (on pp. 602–636), by means of which the author has sought—though not very successfully—to meet the objection that no literature references were given in the first edition to enable the student to follow up any matter of special interest.

One cannot help feeling that the authors of German works on organic chemistry must sometimes be in the habit of saying to themselves "Organic chemistry is a German Science, founded by Liebig of immortal memory," and accordingly ignoring so far as possible the work of all scientists west of the Rhine. This tendency is also noticeable in the section on the cyanine and other sensitising dyes, where the important work of English and American chemists on the subject such as Adams, Mills, Wise and others is altogether disregarded, with the result that several of the formulae quoted by Bucherer stand in need of revision in the light of the most recent research. The list of references already noted is a step in the right direction, but still needs considerable improvement before it can be regarded as satisfactory.

The statement on p. 493 apropos of the decrease in the acreage under indigo cultivation in India during 1916–18 is illuminating:—"Die mit Indigo bebaute Fläche nahm in ganz auffallendem Masse ab, offenbar vor allem infolge der grossen, durch den U-Boot Krieg verursachten Schwierigkeiten, den Farbstoff über See zu verfrachten." We are afraid these lines were penned whilst the eminent German author was still feeling the after-effects of "U-Boot Fieber," and that he will find the cause of the decrease to be a good deal nearer home, say, in the neighbourhood of Elbe and Rostock.

In the chapter on the Natural Dyes the recent work of Dimroth and his pupils on carminic and laccic acids is discussed, but no mention is made of von Lampe's synthesis of Curcumin in 1918 in the section dealing with that colouring matter.

The addition of marginal rubrics makes the second edition somewhat handier than its predecessor, but the use of italics or other distinctive type for sectional sub-headings would be a great advantage. Most of the defects noted are, however, but slight, and the second edition cannot but enhance the reputation already won by the 1914 edition.

F. A. MASON.

ANTHRACENE AND ANTHRAQUINONE. By E. DE BARRY BARNETT. Pp. 436. (London: Baillière, Tindall and Cox. 1921.) Price 27s. 6d. net.

Since the publication, many years ago, of Auerbach's "Anthracen" (1880), a work which is probably unknown to the younger chemists of the present day, no attempt has been hitherto made to collect in book-form an account of the varied reactions of this hydrocarbon and its derivatives. In the interim an extraordinary amount of new matter has accumulated on this subject, much of which is only to be found interspersed among the foreign patent literature, not easy of access to the average student and, moreover, involving search of a tedious character. The appearance of Mr. Barnett's book is thus greatly to be welcomed, and this should prove to be of much service not only to colour chemists, but to students of organic chemistry in general. The volume of approximately 400 pages is divided into eighteen chapters:—I. Introduction; II. Anthracene and its Homologues; III. Simple Anthracene Derivatives; IV. The Anthraquinones and Dianthraquinonyls; V. Anthrone, Anthranol and Allied Products; VI. Anthraquinone Ring Syntheses; VII. The Benzanthraquinones; VIII. The Aldehydes, Ketones and Carboxylic Acids; IX. The Nitro, Nitroso and Halogen Anthraquinones; X. The Sulphonic Acids, Mercaptans and Sulphides; XI. The Amino Anthraquinones and Dianthraquinonylamines; XII. The Hydroxy and Amino Anthraquinones and Ethers; XIII. Pyridine and Quinoline Derivatives; XIV. The Acridones, Xanthenes and Thioxanthenes; XV. The Benzanthrones; XVI. The Cyclic Azines and Hydroazines; XVII. Miscellaneous Heterocyclic Compounds; XVIII. Miscellaneous Compounds; and there are, in addition, addenda and an index of German patents.

The book contains a very large amount of information from the scientific aspect which is well arranged and has without doubt involved considerable labour on the part of the author. On the other hand, one feels that the subject matter is, if anything, of too condensed a character, and might perhaps be more easily assimilated had it been spread over two volumes of about the present size. It appears a pity that in a work of such importance but four and a half pages are devoted to a historical sketch of the subject, and that more attention is not given to the earlier history of the synthetic manufacture of alizarin. References to Schultz and Auerbach hardly meet the case (and, indeed, is not the latter out of print?), for these authors are not entirely unbiased and do not inform one that the successful production of the Alizarin Reds was carried out in this country by Perkin by the dichloranthracene method (a process not alluded to by the author) for some time before they were economically obtained in Germany. Again justice is hardly meted out to the latter, there being no mention of his discovery of anthrapurpurine, 4-amino-alizarin, bromo-alizarin, or of his work on anthraflavic acid and the dihomanthraquinones. Though the book will be found useful for purposes

of reference and, indeed, contains an account of the more recently discovered reactions of importance, there are one or two minor omissions, such as, for instance, the hydroxyanthraquinone- $\beta$ -carboxylic acids,  $\beta$ -methyl alizarin, anthraquinone oxide, etc., though this may perhaps well be the case when there has been so large an amount of material for collection. The statement on page 253 that anthraquinone sulphonic acid on fusion with alkali gives an alizarin containing as impurity higher hydroxylated anthraquinones is hardly correct, as the commercial dyestuff carefully prepared from the  $\beta$ -monosulphonic acid salt contains only alizarin as colouring matter. Again (page 254), though a pure alizarin can be prepared by the alkali fusion of anthraquinone according to the patented methods, it is easy also in this case, if due precautions be not taken, to obtain it contaminated with higher hydroxylated products. It is difficult to see that the employment by the author of his modification of Pfaff's system of notation throughout the book is of advantage, for this does not economise space, and is only likely to confuse the less experienced reader. Frequently, again, these formulae, as for instance on page 318, give one the impression of pentavalent carbon, and it is difficult to understand why a  $\beta$  compounds of this and allied types should be represented by diagonal lines, whereas with the  $\beta\beta$  derivatives parallel bonds clearly indicating tetravalent carbon should be used (p. 317).

Reference to German patent numbers are naturally numerous throughout this work, but it would have been a greater boon to the student had these been either supplemented or replaced by statements as to the volume and page where they occur in Friedländer.

The book is well up to date, bears evidence of careful search by the author, and should prove of assistance to many investigators. It is, however, sad to see among the enormous variety of facts it contains how few have emanated from work carried out in our own country.

A. G. PERKIN.

SILICA AND THE SILICATES. By J. A. AUDLEY. *Industrial Chemistry Series, edited by S. RIDEAL.* Pp. xiv.+374. (London: Baillière, Tindall, and Cox. 1921.) Price 15s. net.

Students on being introduced to the consideration of the industrial applications of silicate chemistry often ask to be directed to some suitable book in English dealing with the subject, and it is not easy to comply with the request. It is possible to recommend works giving descriptions of manufacturing processes, but none which, at the same time, emphasises clearly the underlying scientific principles. The title of the book under review raised hopes that this gap in the literature of silicate chemistry might be bridged, for it immediately suggested parallelism with H. le Chatelier's "*La Silice et les Silicates*." But although the author has drawn on his experience as a ceramic chemist, and on a great accumulation of knowledge, he does not write with the scientific insight and confidence of an investigator nor with any great insistence upon essentials.

The book is divided into six sections, the first dealing with the various modifications of silica, their natural occurrence and artificial production. It is rather surprising to find the regions of stability of the different forms of silica discussed without any reference to the well-known and comparatively recent work of Fenner on this subject, particularly as space is found to mention several old investigations on the artificial production of quartz crystals. At present the stability ranges of the various modifications of silica are debated keenly. They are

by no means definitely established, and the subject calls for very careful treatment if confusion is to be avoided. We find it stated:—

(1) Without any qualification that "tridymite is the stable variety of silica above 1500° C." (p. 36);

(2) that "the temperature is about 1600° C. where the transformation into tridymite takes place" (p. 33);

(3) that cristobalite "thus differs from tridymite both by its birefringence and its point of transformation 400° higher" (p. 34); and

(4) that "Tridymite is not known to be formed under any conditions above 1470° C."

The reader must take his choice. A reference on p. 42 to the impossibility of determining by means of optical investigation whether silica obtained from calcining hydrated silica is crystalline or amorphous might be brought up to date by mentioning the possibility of settling this point by the X-ray method of crystal analysis of powders. Kyropoulos has concluded in this way that amorphous silica shows no evidence of regular, internal structure, but after calcination at 1300° C. the interference bands characteristic of cristobalite appear.

The properties and uses of natural and artificial silicates are discussed in the next part, whilst the third section is devoted to lime, cement, and mortar. Good descriptions of the manufacturing processes are given, but a fuller reference in the text to the diagrams of the various plants would be useful to many readers who are unfamiliar with the industry. This is also true of other sections of the book, almost a complete page being devoted, for example, to diagrams of the Stein tank furnace with no specific reference to it in the text.

The fourth section deals with the ceramic industries, and a very readable account is given of pottery and brick manufacture, together with passing references to more special non-silicious refractories. The earlier remarks with regard to the subordination of the scientific aspect of the subject are illustrated in this section. For example, in a discussion of the fusibility of clays no reference is made to Raoult's law, eutectic mixtures, or to Ludwig's work on this question.

Section five discusses glass and enamels, and considers the raw materials, the composition and properties of various types of glass and enamels, glass furnaces, etc. The last section is devoted to some miscellaneous applications of silicates.

The descriptions of methods of manufacture are clear and concise, and the book will be of use without completely satisfying the demand for an English text-book giving a scientific exposition of the silicate industries. The conclusions of many investigators whose work has appeared in recent years in the journals of the English and American Ceramic Societies and the Society of Glass Technology are summarised. This feature of the book will be very valuable to those who have not the time or opportunity to consult these journals. The references to the results of continental workers are not so numerous. Both the text of the book and the diagrams are well produced, and short bibliographies are given at the end of each section and at the end of the book.

Summarising, this work is full—even crowded—with information both as to the occurrence and properties of raw materials and as to manufacturing processes. It gives also the results obtained by many investigators on many branches of the subject. It loses grip, as do so many technological and scientific books of the present day, by overlaying essentials with more or less relevant detail and in refusing to select or decide—a method of treatment which has both advantages and disadvantages, but is more informing than stimulating.

JOHN W. COBB.

## OBITUARY.

## BERTRAM BLOUNT.

The death of Bertram Blount on April 9, at the age of 54, has removed a well-known member of the chemical profession and of this Society. Educated at King's College, London, under C. L. Bloxam, he started practice with W. H. Stanger, and continued independently when that partnership was dissolved. He devoted himself more especially to engineering chemistry, and in collaboration with A. G. Bloxam published a book on "Chemistry for Engineers and Manufacturers," whilst his individual publications "Cement" and "Practical Electrochemistry" are also well known. On both these subjects he was a widely-recognised authority, and at the time of his death he was editing a series of books on Electrochemistry, two volumes of which have been already published. He was also the author of papers on the "Determination of Oxygen in Commercial Copper," "Vacuum Balance Cases," "The Limitations of the Balance," and other subjects. Blount enjoyed an extensive consulting practice, and was frequently called as an expert witness in chemical law cases. Of marked individuality and characteristic manner, he was not readily forgotten by those with whom he came in contact, and it is a subject for regret that he should have broken down at so early an age.

## EDMUND JAMES MILLS.

Prof. E. J. Mills, who died on April 21 last, was born in London on December 8, 1840. He was educated first at the Grammar School, Cheltenham, and then at the Royal School of Mines, where he took the courses of instruction for a degree in science at London University, at that time the only university in Britain granting such degrees. He graduated B.Sc. in 1863, and obtained the doctorate in 1865. There were few candidates for science degrees at that time, and on looking at the list of graduates it is interesting to note that Mills's name appears about equidistant in time between those of Prof. Alex. Crum Brown, in 1862, and Sir William Tilden, in 1871.

In 1861 Mills became assistant to Dr. John Stenhouse, and a year later was appointed one of the demonstrators in chemistry at Glasgow University, in which capacity he conducted tutorial classes for medical students, a work not very congenial to him, and one which, from his temperament, he was not particularly well fitted; he was, however, a good laboratory teacher for advanced students. He resigned this appointment in 1865. In 1875 Mills became superintendent of the "Young Laboratory," in Anderson's College, Glasgow, and during his term of office, about the year 1886, that foundation, together with Anderson's College, became incorporated in the Glasgow and West of Scotland Technical College, the Young Laboratory appointment being converted into the Young Chair of Technical Chemistry. His predecessors in charge of the Young Laboratory were W. H. Perkin, sen., 1870-71, and G. Bischof, 1871-75.

Mills was the author of many papers covering a wide field of work. Although his first recorded paper in the Proceedings of the Royal Society (1860) is upon brom- and chlor-phenylamine, his early work inclined more to general and inorganic chemistry, dealing with subjects such as isomerism, statical and dynamical ideas on chemistry, chemical equivalents, etc., and this tendency is seen also in his later papers in the Philosophical Magazine, published during 1883, '84, and '87. In the Journal of the Chemical Society his more important papers are:—"On Aniline Derivatives and Nitrotoluene"

(1875-76); "On Potable Waters" (1878-81); "On Chemical Repulsion" (1880-81); "Melting-points" (1882). From 1879 to 1882 he published "Researches on Chemical Equivalence"; from 1878 to 1880 papers on "The Action of Oxides on Salts"; and later, "Researches on Dyeing," in conjunction with Louis Campbell. To the Society of Chemical Industry he contributed papers on the "Quantitative Estimation of Oils and Fats" (1883), "Viscosity Determinations" (1886), and "Oxidation of Aniline" (1894). In his books Mills did not confine himself to purely scientific matters, for, in addition to two works, "Fuel and its Applications" (published conjointly with E. J. Rowan in 1889) and "Destructive Distillation" (1892), he wrote a volume of poems "My Only Child" (1895), and "The Secret of Petrarch" (1904).

Mills was elected a Fellow of the Royal Society in 1874, and was an honorary LL.D. of Glasgow University; he was an original member of the Society of Chemical Industry and served on the committee of the Glasgow Section, including two periods as vice-chairman, from 1884 to 1894; and was a member of the Council of the Chemical Society at various times from 1868 to 1906, serving as vice-president from 1912 to 1915. After retiring from his professorship in Glasgow in the early 'nineties, he returned to London and rendered valuable service as a member of the Library Committee of the Chemical Society.

J. MILLAR THOMSON.

## PUBLICATIONS RECEIVED.

- COKE-OVEN AND BY-PRODUCT WORKS CHEMISTRY. By T. BIRNULPH-SMITH. Pp. 180. (London: Charles Griffin and Co., Ltd. 1921.) Price 21s.
- BIBLIOGRAPHY OF THE ACTIVATED SLUDGE PROCESS OF SEWAGE TREATMENT. By J. E. PORTER. Second edition. Pp. 117. (Rochester, N.Y.: General Filtration Co., Inc. 1921.) Price \$1.00.
- THE CHEMISTS' YEAR BOOK, 1921. By F. W. ATACK and L. WHINYATES. Vol. I., pp. 422; Vol. II., pp. 423-1142. (Manchester: Sherratt and Hughes. 1921.) Price 21s.
- THE ELEMENTS OF VEGETABLE HISTOLOGY. By C. W. BALLARD. Pp. 246. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1921.) Price 18s.
- DIE ELEKTROMETRISCHE MASSANALYSE. By DR. E. MÜLLER. Pp. 110. (Dresden and Leipzig: Theodor Steinkopff. 1921.) Price: paper, 8s. 3d.
- REPORT ON THE SIMMANCE TOTAL HEAT RECORDING CALORIMETER. By T. GRAY and A. BLACKIE. Fuel Research Board, Technical Paper No. 2. Department of Scientific and Industrial Research. (London: H.M. Stationery Office. 1921.) Price 9d.
- THE "BLACK SPOT" OF CHILLED AND FROZEN MEAT. By F. T. BROOKS and M. N. KIDD. Food Investigation Board, Special Report No. 6. Department of Scientific and Industrial Research. (London: H.M. Stationery Office. 1921.) Price 6d.
- BULLETIN OF THE IMPERIAL INSTITUTE. Vol. XVIII. No. 4. October-December, 1920. (London: John Murray. 1921.) Price 3s. 6d.
- WOOD WASTE. By I. H. BOAS. Institute of Science and Industry, Bulletin No. 19. Commonwealth of Australia. Pp. 32. (Melbourne: A. J. Mullet, Government Printer. 1921.)
- THE CONCRETE INSTITUTE. TRANSACTIONS AND NOTES. Vol. IX. (London: The Concrete Institute. 1921.)

## DANGEROUS DRUGS.

As a result of the criticisms which were directed against the draft Regulations issued under the Dangerous Drugs Act (*cf. J.*, 1921, 79 n, 139 n), the Home Secretary appointed a Committee to consider the outstanding objections and to advise on modifications. The Report of this Committee, which has just been issued (Cmd. 1307, 3d.), shows that witnesses were heard on behalf of the British Medical Association, the Pharmaceutical Societies of Great Britain and Ireland, the Drug Club, Fine Chemical Manufacturers' Associations, the Central Chamber of Agriculture, the Association of Public Analysts of Scotland, and twenty-four other allied bodies. After consideration of the evidence, the Committee has recommended certain further modifications and additions which it claims will "almost entirely remove" the objections to the original draft.

In our issue of April 30 last we pointed out that the draft Regulations overlooked the fact that the drugs in question were constantly used in chemical, physiological, pharmacological and other laboratories, and that no provision was made for their possession and use by the experts in charge of such institutions. An attempt, which cannot be regarded as completely successful, has been made to remedy this defect in the revised regulations. In Article II it is stated that " . . . any person in charge of a laboratory for purposes of research or instruction attached to any university, university college, public hospital or other institution approved by the Secretary of State for the purpose, or any person appointed by a local authority with the approval of the Minister of Health as an analyst for the purposes of the Sale of Foods and Drugs Acts, 1875 to 1907," is authorised to be in possession of and to supply the drugs concerned so far as is necessary for the practice of his profession, but subject always to the provisions laid down in the Regulations. Hence scientific institutions which are not "attached" to a university, university college, or public hospital will apparently need "approval" by the Home Office before they can secure a licence, and the same applies to all independent workers such as analytical, consulting and other professional chemists who are not officially-appointed public analysts. This means that such institutions as the Finsbury Technical College, the Imperial College of Science, the Davy-Faraday Laboratory, and the Lister Institute need "approval" before they can possess and use these drugs for research, and that independent workers like the late Sir W. H. Perkin can only secure such a privilege by attaching themselves to a university or a hospital or by becoming public analysts.

Assuming that no further revision will be made, it is to be hoped that the necessary licences will be readily granted to all genuine research workers, and a letter from the Home Secretary to the Parliamentary Committee of this Society seems to suggest that this course will be pursued. In the letter, dated April 27 last, it was stated that the Home Secretary "would be prepared to consider sympathetically all applications for licences thereunder from applicants who require to use the drugs for the purpose of *bona fide* research"; nevertheless, we fail to see the necessity for imposing even such a modified restriction on scientific research and for compelling research workers to submit to the delays and vexations which are invariably associated with applications to Government departments.

There are other considerations which, if of less interest to the majority of our readers, are nevertheless of much importance to our pharmaceutical colleagues.

The Committee could not agree to the request that the requirements of a prescription for the sale of the particular drugs named in the Act should be completely withdrawn, but a list of exemptions has been prepared and inserted as a schedule to the Report. This schedule exempts two well-known and popular remedies—Dover's Powder and gall-and-opium ointment—and fifteen other official and unofficial remedies, none of which, however, is used to any extent in medicine or pharmacy. The Committee ignores the point made by the Pharmaceutical Society that the prohibition is *ultra vires* on the ground that it can only be settled by competent judicial authority. Amendments are proposed to make it clear that dentists, veterinary surgeons, and medical practitioners can administer the drugs to their patients without writing a prescription or keeping a record, and the Pharmaceutical Society's suggestion that the prescription should bear the name and address of the person for whose use it is intended has been adopted. As an attempt to meet various objections from the British Medical Association, the Pharmaceutical Society, and the Drug Club, a new clause has been inserted regarding the marking of packages or bottles containing the specified drugs. Special regulations have been agreed upon concerning the application of the Act to hospitals and similar institutions, but the Committee has been unable to overcome the difficulty that the Regulations do not apply to preparations containing less than 0.2 per cent. of morphine or 0.1 per cent. of cocaine or diamorphine. It was demonstrated that a big bottle containing any one of the drugs specified in the Act of a strength below the limit fixed in the Regulations might actually contain more of the drug than a small bottle of the drug in a concentrated form; so that a person desirous of gratifying the drug habit can avoid the Regulations by purchasing a large bottle of the low strength. The Committee admits its helplessness and states that it cannot be blamed as it did not frame the Act! Again, the Committee fights shy of the submission on behalf of the Pharmaceutical Society (emphasised in a "Reservation" by Mr. W. J. Uglow Woolcock, a member of the Committee, and printed at the end of the Report) that if it is desirable for the protection of the public that a pharmaceutical chemist should be prohibited from dispensing any of these drugs without a medical prescription, it is equally desirable that these drugs should not be dispensed by the doctor who prescribes them.

## LIQUID FUEL FOR STEAM-RAISING.

N. A. ANFILOGOFF.

There is no industry in which fuel oil cannot advantageously replace coal as a source of heat or power. In reverberatory furnaces, metallurgical furnaces, for electric-light-filament carbonisers, cement kilns, rivet forges, and even ordinary cooking stoves, fuel oil has been tried and proved to be more efficient than coal.

For steam-raising liquid fuel is, beyond compare, the more efficient fuel of the two. In the Lancashire boiler, for instance, a third of the length of the internal flues is divided in half by the fire-grates, and no heat at all impinges on the lower half. On the contrary, the cold air entering the ash-pit cools the boiler-plate where the layer of ashes does not insulate it. With fuel-oil firing the whole of the area of the circle of the flue is under heat, thus increasing or making more effective the available heating surface and greatly assisting the

water-circulation. The intensely hot flame, which can be easily controlled, gives a more steady head of steam; and it may be asserted that in any factory where four boilers under coal fires can hardly carry the load, three only of these boilers under oil will suffice. This the writer has personally experienced. One must not forget that one man can quite easily look after four boilers under oil, whereas at least three men would be required if these boilers were hand-fired by coal. There is no cleaning of fires, trimming, or wheeling away of ashes with oil-fired boilers.

During the present coal crisis very many factories adopted oil as "the next best thing" at "whatever the cost," quote forgetting or not knowing that, as a matter of fact, they had been losing money, or, at least, failing to make the most economic use of their boiler-plant, by their previous use of coal.

Apart altogether from savings in many other directions, let us examine how the use of coal compares with that of oil. To-day's prices (or rather pre-strike prices) may be taken as 50s. per ton for coal and 90s. per ton for fuel oil. Examining theoretical values as a guide and taking calorific values, we get an average of, say, 14,000 B.Th.U. for coal and, say, 19,000 B.Th.U. for liquid fuel—that is, 1 of oil is equivalent to 1.4 of coal. Taking also theoretical evaporative values, we get, say, an evaporation of 11.8 lb. of water from and at 212° F. for 1 lb. of coal and 17.9 lb. of water from and at 212° F. for 1 lb. of oil, i.e., in this respect 1 lb. of oil is equivalent to 1.5 lb. of coal. In practice, however, working on actual monthly consumption figures, the writer has hardly ever exceeded an evaporation of 8 lb. of water per 1 lb. of coal, and has invariably obtained an evaporation of 15.6 lb. of water per lb. of oil. These actual consumption figures therefore show that 1 lb. of oil will do the work of 1.95 lb. of coal. Translating these figures into cash values on comparative costs of fuel, we find that 1 ton of coal at 50s. evaporating 8 tons of water gives an evaporation-cost of 6s. 3d. per ton of water, whereas oil at 90s. evaporating 15.6 tons of water gives an evaporation-cost of 5s. 9d. per ton of water converted into steam.

Assuming that we are dealing with a factory employing four boilers, each capable of evaporating 7500 lb. of water per hour, we get the following interesting statement of comparative costs:—

Evaporation 72,000 galls. per 24 hours or 321½ t.

Evaporation cost.	Coal.	Evaporation cost.	Oil.
321½ tons at 5s. 3d.	£100 9 4	321½ tons at 5s. 9d.	£92 8 7
1 Fireman at 2s.	2 8 0	1 Fireman ..	2 8 0
1 Assistant at 1s. 11d.	2 6 0		
1 Trimmer at 1s. 10d.	2 4 0		
	<u>£107 7 4</u>		<u>£94 16 7</u>

Thus there is a clear cash saving of £12 10s. 9d. per day. But without any such saving in cash, the advantages derived from greater efficiency of boiler-plant, ease of control and absence of ash, dust and dirt, in themselves would demand the change from coal to liquid fuel. We are, however, very, very conservative and afraid to make a departure from the methods employed by our fathers and grandfathers.

Speaking to a chemical manufacturer with a large business, the writer was given a reason why the chemical factory was still burning coal that seemed to him to sum up the position—he was told that boilers under coal have lasted 20 years or more, and that no one would come forward to guarantee the life of the boiler under oil. But neither would the boiler-maker guarantee the life of the boiler under coal fires except with the proviso that reasonable care must be taken. Now, in the words "reasonable care" lies also the mystery of success or failure in burning liquid fuel. The work falling on the fireman is so slight and the control of the

liquid fuel fires so easy, requiring, as it does, the turning of a valve-wheel, that there should be no excuse why the most punctilious care should not be bestowed on the boiler.

On the other hand, with oil fuel the whole of the flues of a Lancashire boiler, or the combustion-chambers of tubular boilers, are under equal heat, doors have not to be opened for stoking up, thus admitting gusts of cold air, so that there is no sudden periodic contraction which, under coal-firing conditions, has added not a little to the reduction of "life" in boilers. No reasonable care, however, can be taken of boilers if the man in charge has not sufficient knowledge to qualify him as a stoker, and the writer envies the works manager or engineer of a U.S.A. factory in this particular. No man there can take charge of a boiler unless he has qualified for and gained a certificate or licence, which has to be framed and exhibited in the boiler-room. In this country, unfortunately, anyone can and does "fire" boilers, but that same anyone absolutely must have a little tuition to fit him to look after oil-fired furnaces.

The difference between coal and oil as fuel lies in the fact that whereas in coal we have a fuel of which some 75 per cent. is burnt slowly on the grate as a solid, in oil we have a fuel the whole of which is burnt quickly as a heavy vapour. In the first case combustion must depend on grate-area, in the latter case it depends on combustion-area.

To get the most efficient results from liquid fuel, one must arrange for two essentials:—(1) The provision of means to convert the oil into a heavy vapour; (2) the provision of sufficient air to support combustion.

To convert the whole of the oil into vapour, as the term is ordinarily understood, would, of course, mean that a boiler or still must be provided in which the oil would be converted into vapour by distillation. This, however, is not necessary, as the oil is burnt in practice not as a "vapour," as the chemist understands the word, but rather as a heavy "mist" mechanically created. The instrument used for creating the mist is called the "burner," or "atomiser." The more finely the oil is atomised, the easier it mixes with air, and the easier, therefore, it ignites and burns.

In selecting an atomiser, the intending purchaser is faced with such an array of "systems" and "patent" burners that it is not surprising that he halts bewildered. In this country the best known are the "Walsend-Howden," "Scarab," "White," "Kermode," "Korting," and "Holden." These are not enumerated in their order of excellence. The trouble is that each system is "the best," and the advocates of each insist on the installation of their whole system as absolutely necessary to achieve efficiency. Much nonsense is incidentally made use of further to bewilder the intending purchaser. One or other of the salesmen will impress you that in his system provision is made for superheating the air, which saves fuel and gives better combustion. Of course, air cannot be superheated. It is true that a slight preheating of the air aids the rapidity of combustion, but to use furnace heat, for instance, to preheat the air, and then to return that heat back to the furnace can only be purposeless transfer of heat (minus losses). It can accomplish nothing and can create nothing, just as stirring water cannot increase its quantity.

Then, again, some urge that their burners must work at a certain pressure, and that the oil must be at a certain temperature. This is also nonsense. There is no burner which will work equally well with any oil at fixed pressures and temperatures; so much depends on the composition and viscosity of the fuel itself. In the main there are two systems:—(1) That in which the oil under pressure is



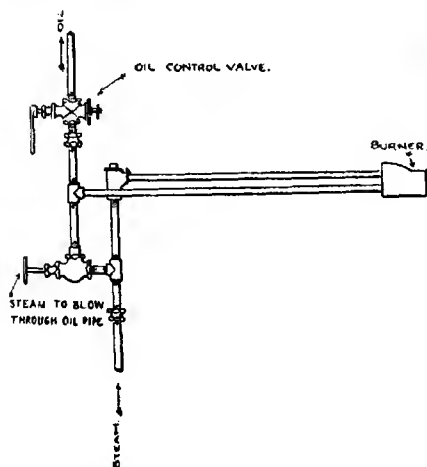
conveyed by more or less tortuous paths to the burner-tip, where it impinges on an obstruction and is forced through a small aperture which causes it to break up into a more or less fine spray; and (2) the system in which the oil is atomised by a stream of air or steam.

On board ship, where water supply is limited, the pressure system is adopted, and for land work, where steam would have to be brought from afar, or where the furnace to be heated is intended for purposes other than steam-raising, the pressure-system would perhaps be the more economical; but for boiler work on land the steam atomiser is by far the easier to handle, the less costly to install, the more efficient from the point of view of smoke-abatment, and certainly not the less efficient thermally.

The installation of the pressure system is very complicated, as it requires a multiplicity of accessories such as filter-chambers, heating chambers, etc., all in duplicate, numerous valves, pipes, and by-passes. The minute orifices in the burner-tip get choked and earthenised, requiring frequent changing, with consequent loss of furnace heat, and the oil used must be of high grade.

In the steam-atomised burner the installation is very simple; any grade of oil can be used, and there are no minute orifices to get choked. But, here, again, there seems to be such a plethora of various "patents" that one perforce has to pause.

There are two kinds of steam-atomisers. In one a stream of oil and a jet of steam meet outside the burner, and the steam, issuing with greater force, carries the oil with it; but to enable the oil to be broken up into a fine spray, a striking-bridge has to be provided inside the furnace against which the mixed stream of oil and steam impinges. These are called "outside mixers" and are more or less efficient according to design, but in atomising consume sometimes as much as 5 to 8 per cent. of the total steam raised.



Burner and Connexions.—Type of "Inside mixer."

The best-designed burner, in the writer's opinion, is the so-called "inside mixer." In this atomiser the oil is delivered to the burner under pressure. The steam is split up into two channels; one conveys the steam into a small chamber, where it actually mixes with the oil and issues from the burner already mixed; the other channel conveys the remaining portion of steam through a slot beneath the mixing chamber, and on issuing from the burner acts as a guide to the mixture of oil and steam.

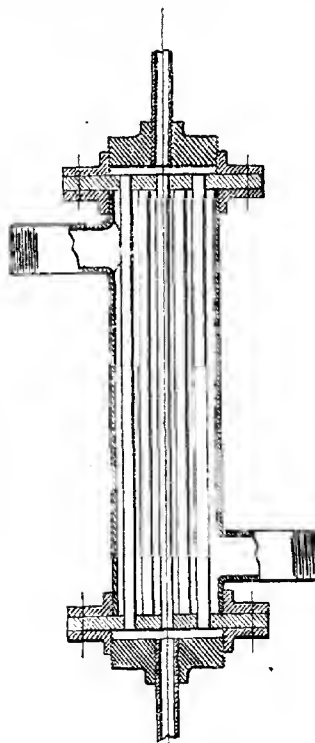
This burner gives a fish-tail flame, like a gas-burner, and burns comparatively noiselessly. In tests over lengthy periods (not a two- or three-hourly test under favourable conditions) the writer has obtained in actual practice thermal efficiencies varying from 79 to 84 per cent., and a steam-consumption for atomising purposes varying, according to load, from 3.0 to 1.9 per cent.

For efficient combustion of liquid fuel sufficient air is, of course, necessary, but one often meets with an insatiable desire to burn liquid fuel absolutely smokelessly. This is a mistake; it is only achieved by using an excess of air. As it is difficult to gauge when just enough air is being used for complete combustion, it is safer to go just short of perfection and allow a haze of smoke to issue from the chimney than to risk an excessive supply of air, with its attendant cooling, unequal distribution of heat in the combustion chamber, and oxidation of boiler plates and burning out of tubes. The writer has a rough and ready formula for calculating the quantity of air required for complete combustion, viz., by dividing the calorific value of the fuel in B.Th.U. by 100, thus:—

Coal, having a calorific value of 14,000 B.Th.U., would require  $14,000/100=140$  cb. ft. of air per lb. of fuel.

Oil, with a calorific value of 19,000 B.Th.U., would require  $19,000/100=190$  cb. ft. of air per lb. of fuel.

These quantities do not fall far short of theoretical requirements and are as nearly as possible actual requirements in practice.



Home-made Oil-heater.  
Scale 1 1/2 inches to 1 foot.

Various text-books and system catalogues show illustrations and describe the necessary firebrick

linings and chequer walls in flues of boilers; the writer has never found them either necessary or even mildly useful.

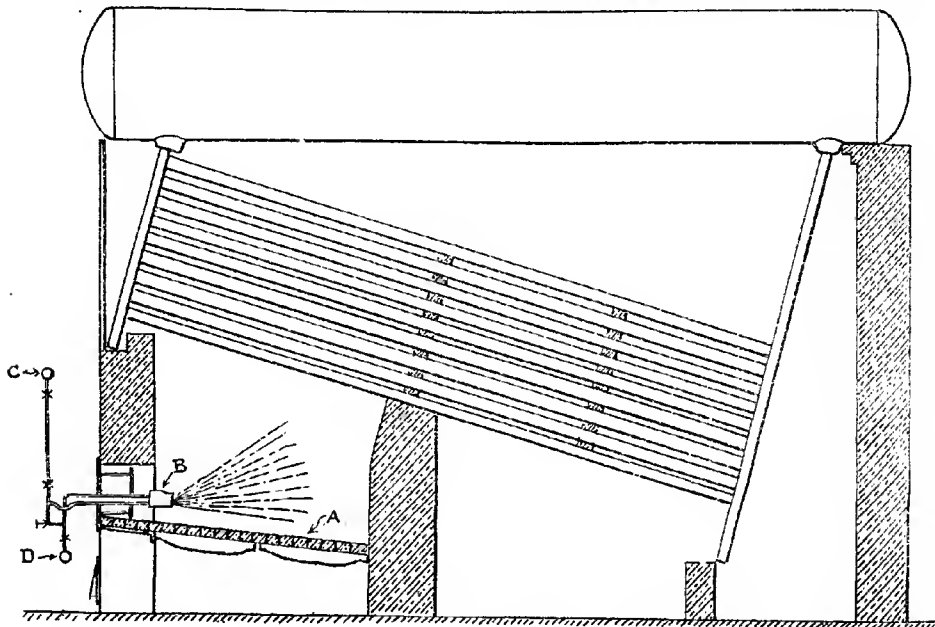
In a Lancashire boiler (except for the striking-bridge required by some atomisers) no firebrick liners are used by the writer, and in the refinery at Thames Haven boilers are at work night and day for six months at a period. After all, the less obstruction there is in the flue the easier does the heat reach the boiler-plate, and the quicker is it conveyed to the water on the other side of the plate.

The Lancashire boiler is converted to oil by taking out the fire-bars, replacing the fire-door by a cir-

thus be effected in one hour at a cost of a few shillings, once the installation is fitted.

In a water-tube boiler of the Babcock and Wilcox type the bars can be left in and covered with firebricks laid flat and dry, i.e., no fireclay is used. The furnace-door is replaced by a plate through which the burner can be inserted, a firebrick wall being built up inside the outer front plate with sight-and-hurner-holes left open. Air is admitted through the hurner-opening and by the ash-pit dampers. The air coming up between the loose bricks lifts the flame off the floor, and high efficiency is obtained.

The "System" itself consists of two small duplex pumps (one as a standby) which suck the oil from

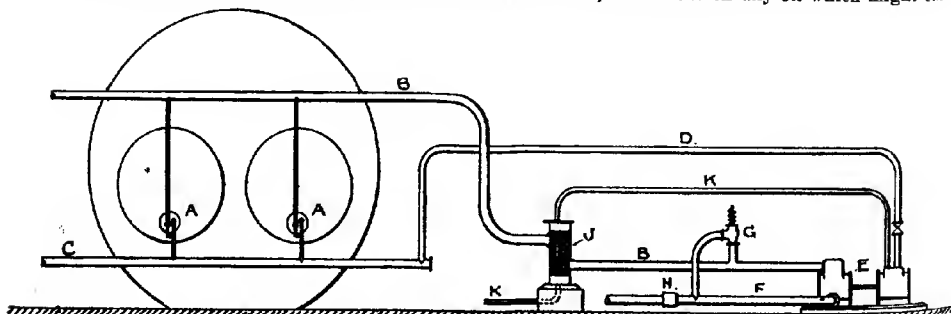


Conversion of a Babcock & Wilcox Boiler to Oil Fuel without alteration of Brickwork.

A.—Firebricks laid on the Firebars. B.—Burner. G.—Oil Supply. D.—Steam Supply.

cular plate through which the burner is inserted, and by providing in the front plate a hinged damper (like an ash-pit damper). A circular cast-iron plate some 3 to 4 in. inside the front plate, or a firebrick wall with chequered bricks (to admit air) is also advisable, as it keeps the flame from buckling the front plate and slightly preheats the air required for combustion. The hurner itself, with its injector-action, draws in nearly all the air required. The conversion from coal to oil or *vice versa* can

the tank and deliver it to the burner. In the suction pipe is inserted a gauze filter in duplicate, and in the delivery pipe a small tubular heater. This is heated by the exhaust-steam from the pump. The pump is also provided with a relief-valve which keeps the charge-line at any desired constant pressure. In the steam-delivery to the burner it is well to provide a valve to enable the oil-ducts to be blown-through with steam before being temporarily shut down, or to clear off any oil which might car-



General Arrangement of Installation.

A.—Burners. B.—Oil Supply Pipe. C.—Steam Supply Pipe. D.—Steam Pipe to Pump. E.—Oil Pump. F.—Oil Suction Pipe from Tank. G.—Relief Valve. H.—Oil Filter. J.—Exhaust Steam Heater for Oil. K.—Exhaust Steam Pipe.

bonise on the hurner tip. It is also wise to provide settling tanks if the oil contains moisture.

To start the boiler from cold, where steam from another boiler is not available, compressed air would be required both to drive the pump and to act as atomiser. This can, of course, be obtained from a small power-driven air-pump or from a storage-cylinder.

When selecting a "System" do not be trapped by claims of "making something out of nothing." Select the simplest. The only desideratum in design should be to develop every heat unit in the fuel and to convey that heat to the boiler with the minimum of loss and without damage to the boiler.

With reference to the oil, it is well to remember that it should (1) have a flash-point (closed test) not less than 150° F., (2) be sufficiently fluid to be pumped at all temperatures (the specific gravity is not always an indicator of viscosity), (3) not contain more than 1 per cent. of moisture, and (4) be free from solid particles of carbon or other extraneous matter which would unduly choke the filter.

Other points of a more general character are:—The boiler-house should be kept clean. There should be no leaks in the oil-pipes of the system. Trays and save-alls should be provided under the pumps and strainers. No oil should be allowed to lie on the floor. Lighted torches should be extinguished before they are thrown on the floor. A water-hose and a sand-box should be provided in each boiler-room. Finally, it must always be remembered that oil heated to or above its flash-point gives off inflammable vapour precisely as petrol does at ordinary temperatures.

## SOCIETY OF CHEMICAL INDUSTRY.

### JUNE MEETING OF COUNCIL.

Owing to the difficulties of railway travelling no meeting was held in May, matters of urgency being dealt with by a special committee appointed for the purpose.

The Council meeting was held on June 3, Sir William J. Pope presiding.

In view of the impending Annual Meeting in Canada the Council had requested the Canadian Sections to suggest one of their members for nomination to the office of president for 1921-22. The Sections proposed Prof. R. F. Ruttan, Director of the Chemistry Department, McGill University, Montreal, and the Council nominated him for election at the Annual General Meeting.

Four vice-presidents retire from office this year (*cf. J.*, 1921, 144 R). The retiring president, Sir William J. Pope, becomes a vice-president, and the Council nominated Mr. J. L. Baker, Mr. C. S. Garland, and Mr. Max Muspratt to the three remaining vacancies. Mr. W. J. U. Woolcock was elected an ordinary member of Council, in place of Mr. W. J. A. Butterfield resigned, for the period ending with the close of the Annual Meeting, 1921; he is, however, eligible for re-election at that meeting. Mr. E. V. Evans was reappointed hon. treasurer, and Prof. H. Louis hon. foreign secretary. It was intimated that June 15 is the last day for receiving nominations of ordinary members of Council.

The balance-sheet and statement of expenditure and income for 1920 were submitted, together with the certificate of the auditors, and these will be published in the issue of the Journal for June 30. Advance copies of the programme of the meetings of the Society to be held in Canada and the United States were laid on the table; copies will be sent to members as soon as a sufficient supply has been received.

It was decided to hold the Annual Dinner of the Society in London early in October; the date and place will be announced as soon as possible.

Forty new members have been elected since the April meeting of Council. The next meeting will be held on Friday, July 15.

### ANNUAL MEETING, 1921.

#### NICKEL PRODUCTION IN CANADA.

During the past three years, despite the many difficulties arising from the abnormal conditions due to the war and its aftermath, a large and important mining and metallurgical enterprise, complete in every detail, has been developed in Canada. The British America Nickel Corporation (through whose courtesy the following information has been made available) has designed and erected mine and smelter plants near Sudbury, Ontario, and a refinery near Ottawa.

The mines owned by the company occupy over 12,000 acres in the mineral-bearing zone, their present activities being confined to the Murray Mine, which so far discloses 16 million tons of ore averaging from 3 to 4 per cent. nickel and copper, sufficient to keep the plants running for 30 years; but the ore body undoubtedly extends much beyond the present operations. In addition to nickel and copper the ore also carries small but valuable amounts of the metals of the platinum group.

The mine plant is thoroughly up-to-date and is furnished with an ample supply of electric power. In the smelter building there are two large blast furnaces in operation with a third in reserve, each having a capacity of over 800 tons of ore per day. The ore, averaging 24 per cent. silica, 35 per cent. iron, 6 per cent. alumina, 3.7 per cent. lime, 4.3 per cent. magnesia, and 19 per cent. sulphur, is smelted without any preliminary roasting. The flux, being converter slag, contains 16 per cent. silica, 52 per cent. iron, 3.5 per cent. lime, and 3.5 per cent. alumina. The charge consists of 70 to 75 per cent. ore, the balance being flux; 10.5 per cent. coke is used with the charge. The resulting slag contains from 0.24 to 0.34 per cent. nickel, iron and copper, together. The low-grade matte, containing about 12 per cent. nickel and copper, is blown in a converter, and the metal content raised to 80 per cent.; the product is then transferred to an oil-fired furnace, in running from which, through a strong stream of water, it is granulated, and in this condition is shipped to the refinery.

At the refinery the matte passes through two Wedge roasters, each with 8 hearths. This is followed by leaching with sulphuric acid which dissolves the copper in part, the metal being recovered by plating out in electrolytic tanks. The cathodes are melted down and cast into ingots weighing about 80 lb., about 55 tons of copper being produced per 100 tons of nickel.

The leached matte is then smelted with fluxes in specially designed electric furnaces. Circular carbon electrodes are used and the nickel copper anodes are cast in steel moulds. The anodes then go to the nickel-depositing building, which covers three acres, where most of the nickel is plated out and a large amount of nickel sulphate and nickel-ammonium salts is produced for the nickel-plating industries. The precious metals are contained in the slimes from the dissolved nickel anodes; they are collected, concentrated, and refined in another department, yielding platinum, palladium, iridium, rhodium, and some gold and silver. Economical power, to an almost unlimited extent, is available from the development at the Chaudière Falls.

Members attending the annual meeting will doubtless be interested in visiting this metallurgical plant, which is probably the largest of its kind in the world.

### CHEMICAL ENGINEERING GROUP.

The second annual meeting was held in London on May 31. Mr. J. Arthur Reavell, chairman, presiding. The hon. treasurer's report called attention to the necessity for increasing the revenue, as during the past year expenditure had exceeded income by £163, or £13 allowing for the sum of £150 set aside to cover the cost of printing the *Proceedings* for 1920. On the credit side, subscriptions amounted to £449 (from 427 members), donations to £327, the grant from the Society to £150; and the main items of expenditure were printing and stationery £507, clerical assistance £343, special and miscellaneous expenses £161. On the motion of Mr. W. Macnab it was agreed to make a levy of 7s. 6d. per member to clear off the deficit. Mr. H. Talbot, in his report, reviewed the activities of the Group during 1920, special reference being made to the success of the conferences held in Birmingham and Newcastle. The Group had done useful work on sub-committees of the British Engineering Standards Association, had co-operated with the Association of British Chemical Manufacturers, and had assisted in opposing the Civil Engineers' (Registration) Bill, which might have adversely affected the position of chemical engineers. Good progress was being made with the compilation and publication of data sheets.

The chairman, hon. secretary, and hon. treasurer were re-appointed, and the following were elected to fill the four vacancies on the committee:—Messrs. W. Macnab, P. Parrish, H. J. Pooley, S. G. M. Urc.

At the annual dinner which followed, Mr. J. Arthur Reavell gave an address on the future of chemical engineering. The present condition of chemical engineering, he said, was less promising than it was a year ago. The very difficult conditions of to-day render it almost impossible for plant manufacturers to make promises regarding delivery, or to put forward with any degree of confidence proposals for new schemes to be undertaken by users. Further, there was very fierce competition from abroad, and buyers were already turning to Germany and to America for plant which they had bought in this country during the war; differences in the rates of exchange were helping our competitors and damaging us.

The answer to the question: What is the position of our chemical engineering industry under such depressing conditions? was that a Britisher was never more dangerous than when everything seemed perfectly hopeless. As we were getting into that position to-day, our future was not so black as it appeared; with the obstinacy and tenacity of our race we should even now be able to put up a fight for the world's trade that would surprise everyone. The future of our industry was closely bound up with the question of the treatment of "key" industries. The Government must decide once and for all whether it is going to protect such industries, or whether it is going to leave our manufacturers and traders to take care of themselves. Meantime, this uncertainty was holding up business. Personally he thought that unless the nations agreed to general disarmament, certain of our resources, which eventually must be used for our own protection in time of war, would have to be protected during times of peace.

The most serious menace to future progress was the unsettled condition of labour. He was convinced from his own observations that the average workman, realising what the extreme labour policy was leading to, was anxious to return to more normal conditions, and this spirit must be encouraged. Until conditions were stabilised, and capital interested, nothing of moment could be accomplished. It was important that men and groups of

men who finance large schemes should co-operate with manufacturers of plant to their mutual profit. The financier, like the manufacturer, looked for the best market for his goods, and it must be a great temptation for him, with the present rate of exchange, to take advantage of foreign purchases. This, however, was no help to either party if in the end the whole country was the poorer because of it. That was one of the most important points that affect the future of our chemical industry. Another important point was ability to put products and plant at competitive prices on the world markets; and in order to minimise cost, there had been of late a rush for standardisation. Standardisation had many great advantages, but it should not be over-estimated. The success of British engineers and chemists had been due to their individuality, so that if the best was to be obtained, industrial effort must not be cramped by carrying it too far. If standardisation were largely confined to parts that are in common use, it would be of help in the future of the industry. Efficiency must be encouraged in every direction; rule-of-thumb working must disappear, and properly trained technical staffs must be available to cope with the ever-changing problems that arise. The future of chemical engineering was bound up with the future of the chemist. The duty of chemical engineers was to put into practice the results of the research and the experiments of the chemist, so that originality of thought and research on the right lines were indispensable to success. Every chemical engineer in this country should rally round the Chemical Engineering Group, so as to make it a huge success. The difficulties with which all young people had to contend were being successfully overcome, and with a better mutual understanding and closer co-operation with the parent Society, the future of the Group and of chemical engineering in this country was assured.

### NEWS FROM THE SECTIONS.

#### MONTREAL.

The annual meeting and dinner were held at the Queen's Hotel, Montreal, on April 22. Mr. C. R. Hazen, chairman of the Section presided, and referred to the regretted death of Dr. Henri St. Georges. The hon. secretary, Mr. G. D. McIntyre, stated in his report that the average attendance at the meetings of the past session had been 55 to 60, and that although a number of members had been transferred to the roll of the new Section at Shawinigan Falls, there was considerable room for improvement in this respect. Four new members and 27 associates had joined the Section during the session. The new draft Rules were submitted and referred to a special sub-committee. On the report of the nominating committee the following officers were elected:—Chairman, Mr. W. H. Matheson (vice-president of Canadian Electro-Products, Ltd., and of Shawinigan Laboratories, Ltd.); hon. treasurer, Mr. T. W. Horner; hon. sec., Mr. J. M. Dickson. The ballot for the election of ordinary members of the committee resulted in the appointment of Messrs. J. B. Bell, R. M. Maclean, G. D. McIntyre, H. J. Roast, and W. B. Woodland; the retiring chairman, Mr. G. R. Hazen, also becomes a member of committee.

#### BRISTOL AND SOUTH WALES.

The report of the hon. secretary states that the membership of the Section is now about 235. Six meetings were held in Bristol, the average attendance being 40, five in Cardiff and one in Swansea. Mr. C. J. Waterfall was elected chairman, in succession to Mr. Walls, and Mr. A. Marsden succeeds Dr. F. W. Rixon as hon. secretary.

## MEETINGS OF OTHER SOCIETIES.

## THE FARADAY SOCIETY.

The soil is such a complex mixture of inorganic and organic materials, and is the seat of so many and varied processes—physical, chemical and biological—that the phenomena exhibited by it require for their investigation the co-operation of the physicist, the chemist and the biologist as well as the active support of the agriculturist. It was therefore a very happy idea of the Council of the Faraday Society to organise a general discussion on "Physico-chemical Problems relating to the Soil" and to invite the presence of members of the Agricultural Education Association, the Ecological Society, the Association of Economic Biologists, and other allied societies. The discussion was held on May 31 at Burlington House, with Sir Daniel Hall in the chair. No less than six of the papers communicated were by past or present members of the Rothamsted staff. The discussion was not, however, confined to British workers; two papers were sent by prominent American investigators, and a special feature of the meeting was the presence and active participation of Prof. Sven Odén, of the College of Technology, Stockholm.

Sir Daniel Hall, in his opening remarks, referred to the big change which had taken place in the viewpoint of soil investigation through the modern development of the colloidal hypothesis.

The discussion was opened by Dr. E. J. Russell, who gave, in a general introduction, an explanation of modern views on the constitution of the soil. The mineral particles that make up the framework of the soil are regarded as being coated with a jelly-like colloidal layer of both inorganic and organic material, which has a far-reaching effect on the properties of the soil. The inorganic colloid contained in the clay fraction and the organic colloid both have a large share in contributing to these effects, whilst the effect of calcium carbonate on the state of the clay also has an important influence on soil texture. The soil solution was next dealt with, stress being laid on the disparity between the plant's needs for water and for nutrient salts, so that the very low concentration of the soil solution (e.g., about 0.002% for nitrates) does not necessarily connote any shortage of nutrient material for the growing plant. The absorptive power of the soil colloids and its influence on the composition of the soil solution and on other special phenomena such as soil "acidity," pan formation, were also considered.

The subsequent papers were divided into four groups. The first of these, dealing with the soil moisture, was opened by Mr. B. A. Keen, of Rothamsted, with a paper on "The System: Soil—Soil Moisture." Work on this subject was critically reviewed, and the various divisions into which soil moisture have been classified were discussed in the light of modern views on the constitution of the soil. The rôle of soil colloids in bringing about a progressive gradation in the states of soil moisture, in contrast to the clear-cut divisions formerly imagined, was explained, the various coefficients such as "moisture coefficient," "wilting coefficient," being regarded as equilibrium points between the different states. Recent American work on the osmotic concentration of the soil solution was considered in this connexion. Prof. Odén then read a short "Note on the Hygroscopicity of Clay and the Quantity of Water adsorbed per Unit-Surface," dealing with an interesting application of his new method of mechanical analysis to the determination of the amount of water adsorbed by clay per unit-surface. This application brings out differences due to chemical nature and degree of

dispersion, whereas the older Mitscherlich method of calculating the surface of the particles from the hygroscopicity took no account of such differences. Two other papers were communicated in this group, by Prof. D. R. Hoagland, of the University of California, on "The Soil Solution in Relation to the Plant," and by Dr. C. A. Shull, of the University of Kentucky, on "Osmotic Phenomena." In the absence of the authors, *résumés* of their papers were read. The first paper dealt with modern work on the reaction and composition of the soil solution, as they affect, and are affected by, the plant. The second paper discussed the osmotic phenomena concerned in the passage of water from the soil into and through the plant. The nature of semi-permeability in general and of plant membranes in particular was considered; Free's theory of protoplasmic permeability and Loeb's suggestion of electric endosmose were specially noted.

The second group of papers dealt with the organic constituents of the soil. The first paper was by Mr. H. J. Page, of Rothamsted, on "The Part played by Organic Matter in the Soil System." After a critical review of the present state of knowledge of the nature and properties of the humic material in the soil, with special reference to humic acid and to colloidal phenomena, the effect of the humic matter on the properties of the soil was considered under the headings of soil texture, soil moisture, soil temperature, and reaction and composition of the soil solution, and it was shown how the known beneficial effects of organic matter on soil fertility can be largely explained in terms of the colloidal properties of the humic matter in the soil. Prof. Odén then read a paper on "The Application of Physico-chemical Methods to the Study of Humus," dealing specially with his own work on the truly acid nature of humic acid, after which Dr. E. J. Salisbury, of University College, London, brought forward the ecological side of the question in a paper entitled "The Vertical Distribution of Soil Acidity in Natural Soils, and its Relation to Organic Constituents." The different factors influencing the variation of hydrogen-ion concentration with depth in natural soils were considered, with special reference to Cornish woodland soils, and in relation to the degree of humification at different depths; the bearing of these results on the distribution of deep- and shallow-rooted plants was discussed.

The third group of papers dealt with adsorption phenomena. Mr. E. A. Fisher, of the University of Leeds, contributed a paper on "The Phenomena of Adsorption in Soils: A Critical Discussion of the Hypotheses put forward." The author made a powerful plea for the more general recognition of the fact that the applicability of an empirical equation such as that of Freundlich to a soil reaction is no criterion of the occurrence of a true adsorption, since such an equation also applies to many reactions which are not adsorptions, and he advanced arguments to show that many of the facts of adsorption by soil are described equally well, and explained more satisfactorily by purely chemical theories developed from that originally advanced by Way. Mr. E. M. Crowther, of Rothamsted, followed with a paper on "Soil Acidity in its Physico-chemical Aspects," in which he dealt with the methods of measuring the so-called "lime-requirement" and the hydrogen-ion concentration of soils; the action of neutral salts on acid soils and the relation of soil acidity to plant growth were further considered. Owing to shortage of time, Mr. C. G. T. Morrison did not read his paper on "Pan Formation."

The final group of papers dealt with colloidal phenomena. Prof. Odén opened with a paper on "Clays as Disperse Systems," describing his new method of mechanical analysis and its application

to the examination of certain Swedish clay deposits. Mr. N. M. Comber, of the University of Leeds, followed with a paper on "The Mechanism of Flocculation of Soils," in which he attributed the abnormal flocculation phenomena exhibited by clay in the presence of alkalis to the conferring on the clay of "emulsoid" properties by a coating of colloidal silicic acid around the clay particles, thus affording a satisfactory explanation of what has hitherto been a puzzling anomaly in the behaviour of clay compared with the other soil fractions. Mr. J. W. Robinson, of the University College of N. Wales, Bangor, then read a paper on "The Physical Properties of the Soil in relation to Survey Work," in which he pointed out the need for simple reliable methods of measuring the physical properties of soils as a means of defining their inherent capabilities, since the ordinary methods of mechanical analysis are often useless except for purposes of genetic classification. Dr. J. W. Mellor's paper on "The Plasticity of Clay" was taken as read in the absence of the author.

Although the time consumed by authors in presenting their papers was, perhaps, unduly long in comparison with the time available for discussion, the Faraday Society is, nevertheless, to be heartily congratulated on the success of the meeting.

On June 1 a delightful day was spent at Rothamsted, where Dr. Russell and his staff spared no trouble to acquaint the visitors with the numerous investigations that are being carried out. The morning was spent in the laboratories and the afternoon in the fields. Of great interest were the experimental plots put down to wheat and barley to test the relative fertilising efficiency of sulphate and chloride of ammonia.

#### SOCIETY OF GLASS TECHNOLOGY.

The May meeting of the Society was held in the University, Sheffield, on May 18, the president, Dr. M. W. Travers, in the chair. The first paper was entitled "The Shrinkage, Porosity and Density of British Fireclays after Firing at 1500° C.," by Edith M. Firth and W. E. S. Turner. Prof. Turner explained that the paper represented a continuation of the work on certain physical properties of British clays which had been described a year ago at Sheffield. The results now presented related to the shrinkage, porosity, and density of the same set of clays between 1400° and 1500° C. In this temperature-range, eight of the clays showed a negative shrinkage (expansion), and two only exhibited a distinct increase in porosity, the rest showing either a small increase or a large diminution. Increased porosity was undesirable, as it was usually a sign of blistering, due to "over-firing." Of the seven clays which showed blistering, five showed the phenomenon only on the surface. Thus 25 out of the 27 clays investigated were usable at 1400°–1500° C., although those which expanded at this temperature would preferably be avoided.

The second paper was by Mr. J. R. Clarke on "The Effect of the Rays of Radium, X-rays, and Ultra-violet Rays on Glass." Seven lime-soda glasses, three of which contained selenium and three cobalt, were exposed to the action of the  $\alpha$  +  $\beta$  +  $\gamma$  rays,  $\beta$  +  $\gamma$  rays,  $\gamma$  rays, X-rays and ultra-violet rays. The first six were coloured brown by the  $\beta$  rays; the intensity of coloration was greatest on the surface and increased with increasing selenium and cobalt content. As the radiation was prolonged, the intensity increased to a maximum, depending on the percentage of colouring agent, and then remained constant. The seventh glass—pure lime-soda—was only affected by the  $\alpha$  rays. None of the glasses was coloured by  $\gamma$  rays, X-rays, or ultra-violet rays with the exposures given. All the glasses

fluoresced when kept in radium emanation, but a "fatigue" effect was observed at about the same time as the maximum intensity of coloration was reached. It is thought that the  $\alpha$  rays are the chief agents in producing fluorescence, though the  $\beta$  rays are also effective. The colouring is concluded to be due to the formation of colloidal particles in the glasses, and it is suggested that the presence of such particles may be explained by the action of the  $\alpha$  or  $\beta$  rays on dissociated ions already present in the glasses.

#### SOCIETY OF PUBLIC ANALYSTS.

Five papers were read at the meeting held in the Chemical Society's Rooms on June 1, Mr. A. Smetham presiding. In the first paper, on "The Composition of Egg Powder," Messrs. F. F. Beach, T. E. Needs, and E. Russell recorded the results of examining so-called egg powders, comparing them with ordinary baking powders. A table of compositions was given, and it was suggested that a standard based on certain specified figures might be laid down for a minimum percentage of actual egg in such preparations. Mr. N. Evers described the colorimetric method of determining hydrogen-concentration by means of indicators, and gave examples of its utility in replacing titration methods in the determination of the "acidity" or "alkalinity" of various products, and of the purity of certain fine chemicals. Mr. F. R. Dodd, in a paper on "The Estimation of Woody Fibre in Cattle Foods," referred to the discrepancies between the results obtained by different analysts working on the same sample by varying processes and suggested a modification of the method to obviate them. The identification of certain fixed oils by the spectrometer was dealt with by Mr. H. C. T. Gardner, who exhibited absorption spectra of a number of vegetable oils and discussed the possibility of identifying such oils by means of absorption spectra. Spectroscopic examination is not sufficient for identification, but evidence of identity can be obtained in certain cases by measuring the positions of the termination of visibility and the extent of visibility of absorption spectra. In the final paper, Mr. J. L. Lizius described the joint use of two indicators in the titration of acids and bases.

#### THE PHYSICAL SOCIETY.

At the meeting held on May 27, Dr. N. R. Campbell read a paper on "A Method of Micro-analysis of Gases, by the Use of the Pirani Gauge." A well-known equation of the form  $\log p = A - \frac{B}{T - T_0}$  expresses the relation between the vapour pressure  $p$  of any substance and the temperature  $T$ . It can be readily deduced that the value of  $\frac{1}{p} \frac{dp}{dT}$  becomes infinite as the vapour pressure approaches zero at the temperature  $T_0$ , a temperature characteristic of the vapour and called the condensation temperature. If, then, a vapour-pressure-temperature curve be determined for a mixture of condensable gases or vapours, abrupt changes of inclination of this curve are found to occur at temperatures corresponding to the respective condensation temperatures of the constituents of the mixture. These temperatures determine the constituents qualitatively, and the magnitudes of the several abrupt increments of pressure serve to determine the composition of the mixture quantitatively. Mixtures containing minute traces of various gases can be analysed by the method, and to this end a suitable type of pressure gauge has been designed by Pirani consisting of a fine, heated wire immersed in the gas at very low pressure—



of the order of 0.01 mm. of mercury. This wire forms one arm of a Wheatstone network, and the three remaining arms are constituted of manganin resistances. It is found that the electrical energy necessary to maintain the heated wire at a definite excess temperature above its surroundings is a function of the pressure of the gas surrounding the wire. The procedure is therefore as follows: the gauge is connected with a side-tube and the gas therein cooled by immersing this tube in various cooling media, most readily obtained by freezing various substances with liquid air. Thus in analysing a mixture containing phosphorus vapour, water, an unidentified vapour from "vacuum" oil, carbon dioxide, hydrogen, and carbon monoxide, the cooling agents employed are freezing mercury ( $-39^{\circ}$  C.), freezing acetone ( $-95^{\circ}$  C.), freezing ethyl alcohol ( $-117^{\circ}$  C.), liquid air ( $-183^{\circ}$  C.). The side-tube is immersed in each of these baths in turn and the gas pressure determined as above. The various constituents of the mixture are thus readily ascertained both qualitatively and quantitatively. The method is capable of wide application, and it may be applied to the quantitative determination of the several higher unsaturated hydrocarbons present in coal gas.

A paper on "The Intensity of X-ray Reflection by Diamond" was read by Sir W. H. Bragg.

#### ROYAL SOCIETY OF ARTS.

On May 30 Sir Kenneth Goadby read a paper on "Immunity and Industrial Disease," the Rt. Hon. J. R. Clynes presiding.

The author classified industrial disease under two main types:—(a) Primary, due to a specific material handled in the occupation, and (b) secondary, non-specific to industry but to which the occupation may predispose. Although the determining factors of most occupational diseases are known, the factor of individual immunity and susceptibility has been neglected. The primary industrial diseases in which immunity and susceptibility play an important part are those due to bacterial infections, diseases caused by actual contact of the hands or body with poisonous material (e.g., TNT, atropine, mercury), and diseases due to dust, fumes, and vapours (e.g., lead, arsenic, paint, dope, etc.). Of the secondary diseases it may be said that they seldom arise from the same causes as those which produce the primary diseases (e.g., tuberculosis among potters is not a consequence of lead poisoning, but of the inhalation of dust particles). Dust, fumes and vapour are responsible for a large proportion of industrial diseases, and individual susceptibility is undoubtedly a significant factor in lead poisoning. The ordinary routine medical examination is of no value for detecting susceptibility owing to the gradual tolerance which is developed by the majority of persons exposed to lead absorption, and which usually occurs within the first three months of exposure. The high incidence of poisoning in the painting trades as compared with white-lead manufacture is difficult to understand, but in many cases it is due to turpentine. The fact that arteriosclerosis (premature degeneration of the blood vessels) with an increase of blood pressure is far more common among painters than among white-lead workers is due to the vehicle in which the paint is used, i.e., mainly to turpentine and its substitutes.

Modern research has shown that the white blood cells are the chief destroyers of living germs and poisons, and that diminished resistance to poison is accompanied by a decrease in their number. In his researches on the composition of the blood in industrial and other diseases, the author has found that in all chronic infections an alteration in the

proportion of white cells may be detected (mainly a decrease in the chief phagocytes), which usually occurs before the outward signs of ill-health are apparent; and that diminution in white blood cells is accompanied by diminished resistance to such poisons as lead, mercury and paint; such persons are particularly susceptible to lead poisoning, etc. He therefore recommends that blood tests (blood pressure and blood counts) be made on persons entering such occupations, so that by eliminating the unfit the incidence of industrial poisoning would be reduced, and some occupations now regarded as dangerous might even be rendered safe. In the painting trade two examinations, one after three and one after six months' employment, would suffice to eliminate susceptible workers. In concluding, the author urged that trade unions should initiate special health committees, not for providing medicine for the sick, but for conserving their capital—health; and in this he was heartily supported by the chairman.

#### THE CERAMIC SOCIETY.

The Refractory Materials Section of the Ceramic Society held a meeting in Paris on May 11, conjointly with the corresponding French society. Four papers were communicated, the first being a report of "Resistance Tests on Refractory Products under Load at Different Temperatures," by Capt. V. Bodin. The author has experimented with a Frémont machine modified for testing metals, using a special spiral-flame furnace, and a recording apparatus fitted to the machine. The curves thus obtained enabled the crushing load to be determined. The deductions drawn by the author are:—(1) Most refractory products, especially those composed essentially of silica and alumina or silica alone, tend to show a decreased resistance to load on heating, the minimum being generally about  $800^{\circ}$  C. (2) The same products when further heated show rapidly increasing resistance, with a maximum usually at about  $1000^{\circ}$  C. For certain clays this maximum may be four times the minimum, or even more. (3) Still further rise in temperature causes a gradual diminution in the resistance, tending towards zero at about  $1600^{\circ}$  C. with all refractory products. (4) In general, all refractory products tend to become plastic or semi-plastic at about  $1200^{\circ}$  C. or higher. The minimum is very important practically, as the whole of the refractory masonry of a furnace passes through the critical range of temperature corresponding to minimum resistance, both during the heating and the cooling; trouble may then arise which has hitherto not been explicable.

It is suggested that, though the fusing temperature of a refractory brick cannot be varied by modifying size of grog, compactness, etc., the minimum resistance may probably be raised by these means and the quality of the brick thus improved. The author concludes that the refractory properties of hauxite are improved by raising the firing temperature, as Le Chatelier found with clay. The maximum has not the same practical importance as the minimum, but if refractory materials could be chosen so that their working temperatures were exactly those of maximum resistances (which are not the same for all products), the working conditions would be very favourable to durability.

The second paper was a preliminary note on "Dinas Bricks of Constant Volume," by Prof. O. Rehuffat. The author found that the specific gravity of Lagonegro quartzite is reduced from 2.65 to 2.25 after calcining for eight hours at about  $1300$ – $1350^{\circ}$  C. He also found that this quartzite contains 0.31 per cent. phosphoric anhydride. On removing the phosphoric anhydride with concentrated nitric acid and calcining the purified rock

for eight hours at 1300–1350° C., the sp. gr. was found to be 2.6. The author also impregnated a very pure primary quartzite of sp. gr. 2.60 (unchanged even after heating for eight hours at 1300–1350° C.) with 3 per cent. of an alkali-glass containing 16 per cent. phosphoric anhydride, and heated the mixture for eight hours at 1300–1350° C. The product had a sp. gr. of 2.27–2.30, and closely resembled the fired Lagonegro quartzite. Having regard to the wide distribution of phosphoric acid in rocks, the author suggests that quartzites which give bricks of constant volume owe their special properties to the presence of small proportions of phosphoric acid.

The next paper was on "Some Difficulties Experienced in Maintaining a Pyrometer Installation in a Works," by Mr. R. S. Whipple. The author attributes want of success in any pyrometer installation nearly always to (1) the choice of the wrong type of instrument, (2) inefficient use of the instrument, or (3) the instrument itself requiring adjustment or correction. Each of these causes was discussed at length.

The last paper, "Notes on American Practice in Refractories," by Mr. W. J. Rees, was the outcome of the author's visit to the United States last autumn. Mr. Rees observed that the outstanding feature of American practice is the well-developed organisation of plant for production at minimum cost. The elimination of hand-labour is a marked feature of the larger plants, and the use of waste heat in tunnel dryers is also noteworthy. Reference was made to a new machine in which, without the application of pressure, the normal operations of the hand-making process are translated into mechanical operations; the machine is tended by unskilled labourers, and the silica bricks produced from it are equal to the best hand-made bricks. The "Dando" ladle brick, which is used to line large basic-steel ladles, lasts 20 to 30 heats, as compared with 8 to 10 heats for high-grade firebricks. The refractoriness of the raw clays varies between cones 16 and 20, and the maximum firing temperature of the bricks is about cone 4. These bricks are made by a dry-pressing process, and the kilns are down-draught, but with eight short chimneys—one between each two fireholes. There is no preliminary drying of the bricks, so the smoking period lasts about nine days and the burning proper a further six days. The manufacture of silica bricks, etc., was described in detail, and developments connected with magnesite and dolomite refractories were discussed.

#### THE CHEMICAL SOCIETY.

At the ordinary scientific meeting held on June 2, Dr. M. O. Forster presiding, Dr. Harold King, of the National Institute for Medical Research, gave an account of "Derivatives of Sulphur in Commercial Salvarsan." The chief points of interest were that the main sulphur-containing impurity which separates when salvarsan is dissolved in methyl alcohol is a mixed arseno-derivative which on oxidation gives 3-amino-4-hydroxy-5-sulphophenylarsinic acid and 3-amino-4-hydroxyphenylarsinic acid, and that at an intermediate stage in the manufacture of salvarsan the generator of this substance can be isolated in the form of what is probably the corresponding sulphinic acid. Therapeutic experiments on experimental trypanosomiasis in mice point to the undesirability of the presence of this sulphur derivative in commercial salvarsan.

Summarising a paper on "Physical Chemistry of the Oxides of Lead. Pt. I.: The Solubility of Lead Monoxide," Mr. S. Glasstone described the preparation of red-brown, yellow, and red forms of lead monoxide by the action of caustic alkalis on

lead acetate solution. The red form probably consists of finely-divided particles of the red, and the yellow form of agglomerated particles of the red. Determinations of the equilibrium solubility and other data indicated that the differences between these three forms of lead monoxide are due to state of division or agglomeration, and not to enantiotropic changes.

A paper on "Latent Heats of Fusion. Pt. I.: Benzophenone, Phenol, and Sulphur," by K. Stratton and Prof. J. R. Partington, was presented by the latter. A weighed amount of the substance is fused by an electric current passed through a heating coil, the material being surrounded by a bath of partly-fused substance at the melting-point. The following values were found, in gram-calories per gram of substance:—Benzophenone 21.7; sulphur (monoclinic) 8.84; phenol 29.06.

Other papers presented in abstract were:—"Researches on Residual Affinity and Co-ordination. Pt. V. Gallium Acetylacetonate and its Analogues": G. T. Morgan and H. D. K. Drew. "Some New Tricyclic Bases": T. S. Moore and I. Doubleday. Prof. K. G. Naik summarised his recent work on dithioketones and dithioethers, and Messrs. C. K. Ingold and W. J. Powell their studies of some abnormal condensations of malonic and cyanoacetic esters with halogenated methanes.

#### PERSONALIA.

Prof. Lemoine, of the Ecole Polytechnique at Paris, has been elected president of the Académie des Sciences.

Dr. William Wilson has been appointed to the London University chair of physics, tenable at Bedford College.

Dr. G. Rutter, deputy-director of research at the Royal Arsenal, Woolwich, has been appointed director of research in succession to Sir Robert Robertson.

Mr. H. Talbot, hon. secretary of the Chemical Engineering Group of the Society of Chemical Industry, has been appointed general manager of the Weisbach Light Co., Ltd.

Mr. K. C. Browning has been appointed professor of chemistry and metallurgy at the Artillery College (formerly the Royal Ordnance College), Woolwich. Prof. Browning was for fifteen years Government Analyst in Ceylon.

On May 9, at Washington, U.S.A., Dr. F. B. Power was presented with a gold medal, conferred by Mr. Henry S. Wellcome, in recognition of his distinguished services to science during the 18½ years he served as director of the Wellcome Chemical Research Laboratories in London. This honour to Dr. Power will be appreciated by his many friends in this country and not least by members of this Society, to whom his work on the Council, the Publications Committee, and the Committee of the London Section is well known.

To the Advisory Committee of the Board of Trade for the Metalliferous Mining Industry, the Secretary for Mines has appointed:—Prof. H. Louis, Dr. J. M. MacLaren, and Mr. T. C. F. Hall (economic geologists); Mr. F. W. Harbord (metallurgist); Mr. T. Falcon, Dr. F. H. Hatch, and Mr. F. Merricks (mining engineers). Sir C. L. Budd is chairman of the Committee, there are twelve representatives of owners of and workers in the iron, tin, lead and zinc-ore mines. Mr. J. J. Burton represents the iron and steel industry, and Sir K. W. Goadby, medical science.

## CORRESPONDENCE.

## AMERICAN METHODS OF BACTERIOLOGICAL WATER CONTROL.

Sir,—I wish to direct attention to some of the criticisms contained in the review of Milton Stein's "Water Purification Plants and their Operation," by Mr. D. B. Byles in the issue of April 30.

The bacteriological tests, which are stated to be somewhat crude, are, in principle, those adopted by the American Society of Bacteriologists, the American Public Health Association, the American Chemical Society, and the American Waterworks Association. The methods in general are particularly suitable for the control of water-purification plants which, in America, are controlled from works laboratories which are almost invariably staffed by trained men. Whilst the amount of work done on each sample will not compare with that done by the well-known organisation of which Mr. Byles is a member, it must be remembered that the smaller works cannot afford it and they prefer to make five presumptive tests for *B. coli* rather than one test carried to the confirmatory stage. Almost every city with a population of 100,000 and over has its own waterworks laboratory and the scientific control of the purification processes is usually far superior to that in English works, with the exception of the Metropolitan Water Board.

The comparison between English and American standards is entirely misleading, for it is quite erroneous to presume that the latter is a relative one. The American standards are absolute, fixed by the experts attached to the Treasury, and specify that the sample shall not contain more than a definite number of *B. coli* per 100 c.c. The standard, unlike our own, has a definite legal standing.—I am, Sir, etc.,

JOSEPH RACE.

Hereford.

May 18, 1921.

Sir,—In reply to Mr. Race's letter, I would point out that the important test of bacteriological purity of a water is its intestinal-microbic content, and that my criticism was directed to two points, namely (i) that the tests themselves were inconclusive in that the media employed (lactose-broth and litmus-lactose-agar) would include many microbes of non-intestinal origin, and (ii) that the quantity of filtered water tested, namely 0.1, 1, and 10 c.c. in the fluid medium and three 1 c.c. plates in the litmus-lactose-agar, does not permit of very great accuracy; in fact, on these tests it would be impossible to deduce "a definite number of *B. coli* per 100 c.c.," which Mr. Race says is the American legal standard unless that number were 10 or more.

With regard to "standards," I am afraid I used the term somewhat loosely and should have said "the method of expressing results." Several recent American publications which have come to my notice have expressed results in the manner indicated in my review, and this was in my mind when I wrote the words Mr. Race refers to.

I am in full agreement with Mr. Race that, on the whole, the scientific control of American water-undertakings is superior to our own, but it must not be forgotten that, with few exceptions, the supplies to our large towns are derived from upland gathering grounds far removed from any source of pollution.

As I stated in my review, much of Mr. Stein's book is excellent, and presumably the author himself would welcome a reasonable amount of well-meant criticism.—I am, Sir, etc.,

D. B. BYLES.

Metropolitan Water Board, London.  
May 24, 1921.

## POST-GRADUATE TRAINING IN INDUSTRIAL CHEMISTRY.

Sir,—Mr. Francis H. Carr's paper on the above subject (*cf. J.*, 1921, 181 B) comes most opportunely at a time when opinion on this vital question has become dulled by natural relief at relaxation from intensive war effort, by lapse of time from the war period, by the knowledge that money will be required to institute schools such as are outlined, and finally by the serious state of unrest latterly manifested in industry. The need of post-graduate training for those destined to take up the practice of industrial chemistry has been emphasised repeatedly by leaders in science and industry, and if the public could only be brought to realise its importance, the Government might be stirred to action. Now is the time to start this training; to-morrow will be too late.

During the war the Government called on the Universities and Technical Colleges for assistance in the direction and control of scientific warfare in all its phases, and it is well known how well that call was met. The Universities and Technical Colleges now call for aid in founding schools which shall be one safeguard against future unpreparedness. Mr. Carr rightly calls for assistance from the Government, but there is great danger that the swing of the "economy pendulum" will hold back the necessary assistance. Immediate steps, however, could be taken by the Government, without any expenditure of money, and the writer wishes to draw attention to the means available in the hope that some concerted action on the part of those concerned may lead to something being done.

During the war the Government built many national chemical factories for the manufacture of explosives. These were exceedingly well equipped and large stocks of spares were accumulated as necessary to the work in hand. Instead of allowing machinery, plant and stores to lie idle at factories, and I refer particularly to material deliberately acknowledged as surplus Government stock or already advertised for sale, the Government might reasonably place at the disposal of those institutions which have already taken up, or are prepared to take up, training in industrial chemistry such plant and stores as are available. For laboratory equipment there are balances, sample crushers and grinders, electric motors, microscopes, and all kinds of general analytical appliances which would be useful. For technical-scale work there are chemical lead sheeting and piping and all appliances for handling and working same, regulus metal in the form of flanges and valves, lead coils; cast-iron and steel tanks of many varieties and sizes, cast-iron and steel piping, coils and flanges; nuts and bolts, pulley wheels, shafting, belting, agitating gear, steel plates, steel drums, cast-iron and steel valves for all purposes, chemical stone-ware tanks, pipes and taps of all varieties, carbons, weighing machines small and large, pyrometers, thermo-couples, indicators and recorders, gauges, acid-ware bricks and tiles, CO<sub>2</sub>- and SO<sub>2</sub>-recorders, Orsat apparatus sets, centrifugal pumps of cast iron, steel and regulus metal, positive-action rotary pumps, electric motors, tools of every variety, fans and blowers, steel stairways, wood stairways, trucks, trolleys, bucket elevators, band conveyers, portable forges, welding sets, steam boilers, aluminium ware, and enamelled iron ware.

Naturally, if some of this material were thus made available gratis to the Universities and Technical Colleges the prospective money value would be lost to the Government, and estimates of a possible credit from surplus stores, etc., would have to be adjusted to the extent of a few thousand pounds. However, such credit estimates are only of transient value and it would be well worth while

for the country to exchange some of them, at least, for the solid value which the surplus material would acquire when put to such training purposes as outlined by Mr. Carr. Further, the Government has on hand many surplus buildings of wood, urallite, and corrugated iron. Could not some of these be offered as temporary buildings for those institutions which are not already provided with accommodation for technical chemical work?—I am, Sir, etc.,

Edinburgh University.  
May 25, 1921.

A. COTTRELL.

## NEWS AND NOTES.

### CANADA.

**Projected Petroleum Refinery at Fort Norman.**—It is reported that a small refinery is to be built near the Fort Norman oilfields on the Mackenzie River, British Columbia (*cf. J. J.*, 1920, 435 n), and that the material for the plant has already arrived. It is intended to produce petrol for the local aeroplane and river-transport services.—(*Oil, Paint and Drug Rep.*, May 16, 1921.)

### JAPAN.

**Reported Discovery of a Phosphate Deposit.**—According to the *Japan Times*, the "Rasa Phosphate Ore Co.," of Tokyo, has discovered a phosphate deposit on a hitherto unknown island lying to the south of Rasa Island which is superficially estimated to contain 15 million tons. Development work has already begun and the company is negotiating with the banks for a loan of 7 million yen (about £700,000) to finance operations.—(*Chem. Ind.*, May 23, 1921.)

### AUSTRALIA.

**Osmiridium Production in Tasmania.**—Practically the whole of the Australian production of osmiridium is derived from Tasmania, where the output in 1920 amounted to 2000 oz., valued at £77,114, the average price being £38 per ounce. At present there is very little market for the product and the Tasmanian Government is investigating the situation. For some years back large quantities of Tasmanian osmiridium have been used in the natural state in the manufacture of the nibs of fountain pens.—(*Bull. Dept. Tr. and Co., Can.*, May 16, 1921.)

### SOUTH AFRICA.

**Industrial Notes.**—Recent mining developments include the exploitation on a large scale of the asbestos deposits in the Kuruman and Barkly West districts by a new company with a capital of £150,000, and the working of certain nickel and talc deposits in the Barberton district (*cf. J. J.*, 1920, 271 n, 304 n) by the South African Nickel and Talc Mines, Ltd. (capital £100,000).

Among other industrial developments are the establishment of oil mills at Congella, Durban, by the African Oil Mills, Ltd., and the exploitation of certain fibres in the Cape Province for the manufacture of paper pulp and of jute substitutes by a Johannesburg syndicate.—(*S. Afr. J. Ind.*, Mar. Apr., May, 1921.)

**Sugar Production in Natal.**—It is estimated that the sugar produced in Natal during the 1920-21 season will not exceed 150,000 tons of manufactured sugar, as compared with 185,000 t. in the preceding season. But for the severe drought with which the season opened and the excessively wet period at its close, the yield would probably have been higher by

about 50,000 t.; at Illovo, from 12 to 13 t. of cane was required to make a ton of sugar. The production of sugar in Natal during recent years has been as follows:—1916-17, 114,580 t.; 1917-18, 107,000 t.; 1918-19, 155,000 t.; 1919-20, 185,000 t. Formerly sugar-cane was usually planted on large holdings by the owner of a mill, but the present practice is for small holders to plant the cane and sell it to the mills.—(*U.S. Com. Rep.*, Apr. 14, 1921.)

### BRITISH INDIA.

**The Sesamum Industry.**—The final estimate of the sesamum crop for 1920-21 is 368,000 tons over 4,291,000 acres, compared with 449,000 t. and 4,254,000 acres in 1919-20. In addition, sesamum is grown in certain other districts, mainly in Burma, the acreage of which has averaged 1,214,000 and the crop 104,000 t. during the last five years. (*cf. J. J.*, 1921, 87 n.) Exports to countries overseas have been as follows:—1916-17, 84,181 tons; 1917-18, 16,193 t.; 1918-19, 2384 t.; 1919-20, 49,325 t.; and 1920-21, 11,832 t.—(*Ind. Tr. J.*, Apr. 22, 1921.)

**Exports of Minerals and Metals in 1920.**—The Board of Trade has received the following statistics from the Government of India relating to exports of metals and minerals from India during 1920:—

	Long tons.
Brass, bronze and similar alloys ..	128.85
Chromite ..	30,675
Copper ..	79.65
Ferro-manganese ..	3,877
Iron, pig ..	44,868
.. other sorts ..	1,354
Lead, pig ..	21,477.75
.. other sorts ..	12.6
Manganese ore ..	711,424
Tin ..	3.3
.. ore ..	704
Wolfram ore ..	2,934
Zinc or spelter, all sorts ..	75
Metals, unenumerated ..	112.45
Ores ..	1,096
Mica ..	3,825.85
Monazite ..	846
Jade stone ..	221.8
Coal ..	1,228,408
Mineral oils, benzine .. (galls.)	21,240,878
.. other kinds .. (galls.)	823,214
Salt ..	827
Gold (uncoined) .. (oz.)	900,069
Silver .. (oz.)	1,644,315

—(*Board of Trade J.*, May 12, 1921.)

### UNITED STATES.

**Synthetic Resins from Furfural.**—Now that cheap methods of making furfural from corn cobs have been perfected, it is probable that synthetic resins will be made from it on a commercial scale. These resins soften between 25° and 100° C., and are insoluble in water but readily soluble in benzene, acetone and alcohol; they impart a glossy surface to materials to which they are applied.

**Syrup from Sweet Potatoes.**—The Department of Agriculture is erecting a semi-large scale plant for the production of a table syrup from sweet potatoes, which is said to be very satisfactory. Sweet-potato flour has not been a success thus far and new methods for its preparation are under consideration. The boll weevil is responsible for the extension of the cultivation of the sweet potato in localities where cotton is no longer profitable.

**Recovery of Waste Products in Beet-sugar Manufacture.**—A plant for the recovery of waste products from a beet-sugar factory is to be erected at a cost of \$104,000. The amounts of these products to be treated are beet-carrying and wash water 2,000,000 gallons, diffusion-battery and pulp-press water 200,000 galls., lime press-cake containing 50 per cent. water 37 tons, and Steffens' house-wastes 200,000 galls. It is intended to recover potash, to increase the yield of sugar, and to discharge the purified effluent into the river.

**Magnesite in 1920.**—The total sales, and the approximate production, of crude magnesite in the United States in 1919 amounted to 156,266 short tons. During the early part of the year sales were limited owing to the decreased demand from the steel industry which was expecting the arrival of cheaper European magnesite, but this did not happen and buying was resumed in the summer. A Bill was presented to the House of Representatives in 1919 providing for protective duties on imported magnesite, but this was opposed by manufacturers of refractory products, and the Bill had not been passed by January, 1921. Practically all the magnesite produced by the three companies operating in Washington in 1919 was converted into ferro-magnesite and sold to steel plants or makers of refractories; and most of the output of the 18 producing mines in California was used in the manufacture of oxychloride or Sorel cement (finely-ground calcined magnesite mixed with a solution of magnesium chloride and certain filling materials). The use of magnesite cement for flooring and wall plaster is extending. Imports of magnesite amounted to 15,852 short tons, of which Canada supplied approximately 51, Austria-Hungary 17, Mexico 16, and Italy 16 per cent. (*cf. J., 1919, 450 B.*)—(*U.S. Geol. Surv., Mar. 7, 1921.*)

**Lithium Minerals in 1919.**—According to the United States Geological Survey, the output of lithium minerals in that country was 6287 short tons in 1919, or more than ten times greater than in 1916. South Dakota and California supply practically the entire output and the deposits are ample to supply requirements in the United States for a long time to come, even if unexpected uses should be discovered. The chief lithium minerals are lepidolite, a lithium mica obtained largely from California, spodumene, a lithium-aluminium metasilicate from South Dakota, and amblygonite, a phosphate and fluoride of aluminium and lithium, also from S. Dakota. A lithium mineral is also won in Spain at a single mine in the province of Cáceres, which produced only 11 t. in 1918. Much of the lithium minerals produced is manufactured into lithium hydroxide, which is used in a certain type of storage battery, lithium salts and lepidolite are used in glass hatches to reduce the viscosity of the glass (*U.S. Pat. 1,261,015; J., 1918, 418 A.*), and the fluorine in the mineral as an opacifying agent for opal glass. Lepidolite might also be used to reduce the viscosity of enamels, but so far as is known it has not been utilised for this purpose. The bromide and iodide have been used in photography and the cyanide in X-ray work; the chloride is used to some extent in pyrotechny; and the carbonate, mixed with carmine, is used as a nuclear stain. Coal-tar products have largely superseded lithium salts in the treatment of rheumatism and allied affections.

**Sodium Compounds in the United States in 1920.**—The following preliminary figures of production, importation and exportation of sodium compounds in the United States in 1920, compiled by the U.S. Geological Survey, have been supplied by the Department of Overseas Trade (from the *Journal of Commerce, N.Y.*). Sales for the year amounted to 9,886,020 short tons, valued at \$139,336,338, which represent an increase of 8 per cent. in quantity and 17 per cent. in value compared with 1919. Considerable decreases took place in the sales of sodium bichromate, ferrocyanide, nitrate, thiosulphate, and sal soda, but there were large increases in sales of bicarbonate, bisulphate, phosphate, borate, and soda ash. Sodium compounds derived from natural sources (excluding common salt) increased to 42,683 t., valued at \$1,513,179. Imports of sodium compounds also increased, but only in the case of sodium nitrite did importation exceed production. The exports, chiefly to Japan, Canada, and Mexico, are believed to constitute a record.

#### Sales of Chief Sodium Compounds, 1919-20.

	1919.	1920.
Sodium acetate .. .. .	778 ..	1,020 ..
Sodium bicarbonate .. ..	134,962 ..	188,906 ..
Sodium bichromate .. ..	26,526 ..	25,973 ..
Sodium bisulphite .. .. .	11,819 ..	22,059 ..
Soda ash .. .. .	981,054 ..	1,242,490 ..
Sal soda .. .. .	80,090 ..	62,837 ..
Sodium hydroxide .. .. .	311,388 ..	362,680 ..
Sodium phosphate .. .. .	14,760 ..	30,515 ..
Sodium silicate .. .. .	306,138 ..	304,503 ..
Salt cake .. .. .	134,685 ..	184,948 ..
Glauber's salt .. .. .	42,087 ..	44,479 ..
Nitre cake .. .. .	88,402 ..	308,688 ..
Sodium sulphide .. .. .	45,448 ..	42,952 ..
Sodium tetraborate .. ..	28,518 ..	35,281 ..

#### Imports.

	1919.	1920.
Sodium chlorate .. .. .	262 ..	281 ..
Sodium cyanide .. .. .	1,208 ..	3,795 ..
Sodium nitrate .. .. .	1,024,053 ..	1,480,519 ..
Sodium nitrite .. .. .	4,570 ..	4,845 ..

#### Exports of Domestic Products.

	1919.	1920.
Soda ash .. .. .	50,481 ..	8,338 ..
Sal soda .. .. .	5,553 ..	6,015 ..
Caustic soda .. .. .	82,113 ..	112,069 ..
Sodium silicate .. .. .	12,150 ..	17,048 ..

Better prices for sodium compounds were obtained in 1920 than in 1919, although they fell off towards the end of the year. Sodium sulphate was in great demand from abroad. Prices of the raw materials used in the manufacture of sodium compounds now appear to have come down almost to pre-war levels.

#### GENERAL.

**British Laboratory Ware.**—The question of the quality, supply, and prices of British laboratory glassware, porcelain, and chemicals, including research chemicals, is being considered by a committee of the British Science Guild, of which Sir Richard Gregory is chairman. In view of the conflicting statements which have appeared from time to time on these matters, the committee is anxious to obtain the views of scientific workers who have experience of recent articles of the kind described, both of British and foreign manufacture. It is obvious that the information can be of use only when it applies to goods of definitely known origin. The points on which information is desired are:—The quality of the goods; their price as compared with that of imported articles of the same quality; the facilities for obtaining supplies; and the effects, if any, on research work of restrictions imposed on the importation of German goods. The committee would also welcome statements made, or reasoned conclusions arrived at, by competent bodies which have investigated these questions recently, and from manufacturers who wish to add any further definite information to that which has already appeared in the Press. The information should be sent as soon as possible to the secretaries of the committee, Prof. J. R. Partington, East London College, or Mr. C. L. Bryant, 23, Peterborough Road, Harrow.

**Carbon Monoxide Poisoning.**—With the object of obtaining accurate data concerning the after-effects of inhaling carbon monoxide, and their influence on loss of working time, a systematic survey has been made in eight States of America of cases occurring in metalliferous mines, coalmines, engine-rooms, and in industries using blast furnaces and producer-gas boilers. It was found that carbon monoxide rarely causes late after-effects following acute gassing and that when such effects do appear, there is nearly always evidence of a pre-existing pathological condition. Frequent exposure to carbon monoxide causes headache and malaise, but no evidence of a cumulative harmful effect was obtained;

permanent compound with hæmoglobin. A certain tolerance to the gas can be acquired, owing to the compensatory increase of hæmoglobin and red cells, induced by prolonged want of oxygen. An advance in treatment has been made by adding carbon dioxide to the oxygen inhalations administered, recovery being three times as rapid as when oxygen is used alone. The U.S. Chemical Warfare Service has perfected a portable respirator which is effective, even at 0° C. against 1 per cent. of carbon monoxide; its life is approximately three hours.—(*J. Ind. Hygiene, May, 1921.*)

"Zinc (1913-1919)," issued in pamphlet form (112 pp., price 3s. 6d. net) by the Imperial Mineral Resources Bureau, consists of a digest of statistical and technical information relative to the production and consumption of zinc in the British Empire and foreign countries. The fact is emphasised that within the Empire are to be found some of the largest deposits of zinc ore in the world, viz., Broken Hill, Australia, Bawdwin Mines of Burma, Rhodesian Broken Hill and British Columbia. Yet, prior to the war, these ample resources of raw material were little utilised by British smelters. In England, ores imported chiefly from Spain, Algeria and Sardinia were mostly used. The only plant treating Broken Hill concentrates was that at Seaton Carew, where the concentrates are roasted and the zinc distilled, sulphuric acid being recovered as a by-product; and the only smelter plant in the Empire, other than in England, was at Port Pirie, Australia.

The usual valuable bibliography is given to cover the period, and an interesting feature of the publication is an appendix occupying seven pages on "The Zinc Industry at the close of 1919," by Mr. Gilbert Rigg of the Broken Hill Associated Smelters. Mr. Rigg, who discussed the subject from both the technical and economic points of view, expresses the opinion that, although many improvements had been effected in the distillation process resulting in increased capacity, fuel economy, and improved recoveries, the electrolytic process, perfected and developed during the war by such companies as the Anaconda Copper Co., U.S.A., and the Trail Consolidated Smelters, Canada, that were previously in no way concerned with zinc production, had come to stay. On the other hand, the electrothermic process can be considered as hardly taking rank with the distillation and electrolytic methods, the successful condensation of the zinc vapour being even more difficult and complicated than in the distillation process.

**Industries in Egypt.**—Although essentially an agricultural country, Egypt possesses a few manufacturing industries and the Government is doing all it can both to strengthen those already existing and to foster new ones. A Government Bureau of Commerce and Industry has been organised for the promotion of native industries, and the Department of Technical Industry and Commercial Education controls 17 trade schools which are turning out skilled manual workers. Among Egyptian industries, sugar-refining takes first place. This industry is monopolised by a company with a capital of 114 million francs which owns a refinery with an annual capacity of 100,000 tons of sugar. During 1918, 79,488 t. of sugar and 37,330 t. of molasses were produced from 814,842 t. of cane. Alcohol is produced from molasses at a distillery with a capacity of about 2,200,000 gallons per annum. An export trade in alcohol was built up prior to 1917 when exportation was prohibited. Small quantities of such by-products as potassium carbonate, chloride and sulphate, and carbon dioxide are produced, and sugar-straw is manufactured for use as fodder. The cottonseed-crushing industry is of some importance, but only local

requirements are supplied; the equipment of the mills is mostly modern and an oil of good quality is produced for edible purposes or for the manufacture of a low-grade soap. Tanning and leather-working are widely practised, pottery is still a flourishing industry, and the manufacture of cement is increasing in importance; bricks and tiles are made at El Widi, south of Helwan, and an impure fireclay is worked at Assuan. Factories for the manufacture of paper, rubber, etc. have been established in recent years, and brewing as well as the distillation of potable spirits is carried on. The most important mineral product is petroleum, the output of which has risen from 12,786 t. in 1913 to 281,885 t. in 1918. Hurgada is the chief producing centre and the oil is refined at Suez in a plant with a daily capacity of 1200 tons. In view of the growing importance of oil fuel, further prospecting work is to be carried out. The mining of phosphate rock is also of growing importance.

**Cost of Fixed Nitrogen in Norway.**—T. C. Hagemann, consulting engineer to the Norwegian nitrate industry, Christiania, has given the following approximate figures of costs for the arc process of nitrogen fixation in order to show that this process is profitable in Norway.

Assuming a yield of 540 kg. of nitric acid (calculated at 100 per cent. per kw.-yr., or 62.5 g. per kw.-hr.), a medium-sized factory (about 40,000 kw.) in Norway will produce yearly about 20,000 tons of nitric acid, equivalent to 4500 t. of nitrogen, in the form of 29,000 t. of Norwegian saltpetre (13% N) and 4400 t. of sodium nitrate (16.5% N). The approximate running expenses are (krone=1s. 1½d. at par):—

Power, 40,000 kw. at 12s. 6d. .. ..	£ 225,000
Administration and wages .. ..	84,375
Raw materials, etc. .. ..	61,375
Packing .. ..	56,250
Sundries .. ..	22,500
Total .. ..	£450,000

The raw materials required are limestone 15,000 t., lime 500 t., and soda 2500 t. The market price of the nitrogen in the above-mentioned products is about £168 15s. per ton, so that 4,500 t. would sell at £759,375. The profit, therefore, is £309,375, and assuming that the capital invested be about £1,125,000 and amortisation charged at 7½ per cent., the total net profit would be £225,000. As power represents half the running expenses, this item, together with the future yield, is vitally important when considering the cost and returns of any individual factory. Calcium nitrate is now being exported from Norway in increasing amounts. —(*Chem and Met. Eng., March 23, 1921.*)

**The Sulphur Industry in Sicily.**—The year 1920 was marked by considerable increases in the production and exportation of Sicilian sulphur; production rose from 181,744 to 240,857 metric tons, the exports increased from 147,286 t. to 188,865 t., and stocks at the end of the year were 147,900 t. as against 137,100 t. in 1919. Relatively good as these figures are, they compare hardly with those for 1914, when 340,700 t. was exported and stocks mounted to 359,800 t. Exports to France have decreased from 60,700 t. in 1914 to 37,100 t. in 1920, to Austria from 25,300 t. to 700 t., to Germany from 18,800 t. to 4,700 t., to Russia from 21,200 t. to 2,200 t., to Scandinavia from 25,200 t. to 4,600 t., and to England from 12,900 t. to 11,600 t. On the other hand exports to Spain and Portugal have increased from 21,400 t. to 30,700 t., and to South America from 900 t. to 8000 t. Exportation has naturally been affected by the fluctuating exchange, but the increasing competition from Louisiana sulphur will, it is anticipated, bring about a reduction in prices.—(*Chem. Ind., Apr. 25, 1921.*)



**Salt-petre Deposit in the Sahara.**—The scarcity of salt-petre in France during the war induced the French Government to send out a technical commission in 1917 and 1918 to investigate the salt-petre deposits situated in the Sahara between Sba and Ouled-Mahmud. Disappointing results were obtained by the Commission, the reserve of salt-petre earth being estimated at only 900 tons. About 60 tons of salt-petre was obtained from this source during the war with an average composition of  $\text{KNO}_3$  53–55%,  $\text{NaNO}_3$  31–33%,  $\text{NaCl}$  2–6%, sodium, calcium, and magnesium salts being also present in small amounts. In view of the small value of the deposits the Commission, which continued its investigations until November, 1919, has advised the French Government to discontinue development work. The product differs from Chile salt-petre chiefly in its high but fluctuating content of potassium nitrate.—(*Chem. Ind.*, Apr. 25, 1921.)

**Electrolytic Sodium Perborate.**—According to a recent patent of the Norwegian firm Fredrikstad Elektrokemiske Fabriker, the process for manufacturing perborate by electrolysis has been simplified and rendered more efficient by employing an electrolyte containing, besides borax, a certain amount of sodium cyanide. Hitherto, concentrated solutions of borax and a large amount of sodium carbonate have been considered necessary for success and the presence of certain metallic impurities has diminished the current efficiency. These difficulties, it is claimed, have been surmounted, so that a dilute solution of borax may be employed, and the presence of sodium cyanide in the electrolyte prevents metallic impurities from bringing about the decomposition of the perborate and thus diminishing the yield. The process appears to be a marked improvement on those already introduced for the manufacture of this valuable per-salt.—(*J. of Commerce, N.Y.*, Apr. 21, 1921.)

**Mexico in 1920.**—The Ensenada district of Mexico includes a large part of Lower California where there are large undeveloped deposits of gold, silver, copper and iron. Copper ore is produced at the El Boleo mine near Santa Rosalia, which is owned by French and Dutch interests and is said to have supplied over 100,000 tons of metal since it was started in 1887. At the present time seven reverberatory furnaces and a 150-ton oil furnace are being used. The Alamos gold district, formerly very important, is being re-opened, silver is being mined near La Paz, magnesite near Magdalena Bay, manganese near Mulege, and tungsten at Real del Castillo. Near Santa Catalina there is an onyx quarry said to be the largest deposit of the kind. Petroleum has not been found in Lower California, but indications are said to be favourable and American, British and Mexican interests have acquired concessions. Another product of this district is sea-bird guano, of which 478 tons was exported in 1919 and 234 t. in 1920. The only resources of the Frontera district are agricultural and forest products; in 1920 the production of cacao amounted to 1473 t., the largest crop since 1913, and that of unrefined sugar to 3000 t. The possibilities of sugar production in the State of Tabasco are very great; and two companies are prospecting for oil, which is believed to occur in large quantities. Henequen fibre is the chief industry of the Progreso district and constitutes about 98 per cent. of the total exports. Although 154,248 t. of fibre was exported in 1920, the actual production was about 126,298 t. The Vera Cruz district is essentially agricultural, producing coffee, vanilla, chicle, and hides. Owing to the bad weather the vanilla crop is expected to be only one-half of that of last season's (about 142 t. of beans). The crop of chicle is very poor, and the production of hides has been much reduced by the destruction of livestock during the revolutions.—(*U.S. Com. Rep.*, Mar. 23, 1921.)

## PARLIAMENTARY NEWS.

### HOUSE OF COMMONS.

#### *Paper-making Industry.*

In a written answer to Rear-Admiral Adair, Sir P. Lloyd-Greame said that at present many paper mills were either closed down or working short time, owing to the high cost of raw materials, the increasing amount of paper imported, and the decline in demand. In so far as foreign competition was from countries with depreciated currencies, no industries other than food industries would be excluded from the provisions of Financial Resolution No. II., relating to the safeguarding of industries.—(May 24.)

#### *Oil-boring in Great Britain.*

Replying to Major Kelly, Sir R. Horne said that of the eight boreholes completed, one was producing oil in commercial quantities and the remaining seven were being closed down; three other borings were still in progress. The date of termination of the agreement with Messrs. S. Pearson and Sons, Ltd., depended largely on the settlement of the question of the ownership of the petroleum discovered, in respect of which legal proceedings were pending. In the meantime, expenditure was being reduced.—(May 24.)

#### *Coal Imports.*

Mr. Bridgeman, in answer to Mr. W. Thorne, stated that the import of coal into the United Kingdom in 1920 amounted to 3671 tons, of an average c.i.f. value of 145s. 1d. per ton. During January, February, and April, 1921, the imports were 142 t. (80s. 5d.), 5591 t. (47s. 8d.), and 2818 t. (58s. 2d.), respectively. No coal was imported in March, 1921.—(May 26.)

In reply to Mr. J. Guest, Mr. Bridgeman said that since the stoppage of work in the mines 94,000 tons of German reparation coal had been imported into this country. He was not aware that the German Government had protested against the exportation of this coal by France and Belgium.—(June 1.)

#### *Subsidies to Rubber Planters.*

In reply to Sir W. de Frece, Mr. Churchill said that he understood that certain foreign Governments granted a subsidy to rubber planters under their jurisdiction. He was not aware that a similar action was contemplated in any British Colonies.—(May 31.)

#### *Home-Grown Sugar, Ltd.*

Answering Mr. Wise, Sir A. Boscawen said that the value of the property, including the factory, estate, farm stock, etc., of Home-Grown Sugar, Ltd., was £447,659 on March 31 last. Shares had been issued to between 500 and 600 shareholders to the amount of £500,000, of which £498,415 15s. had been paid up. There were no debentures, but the company was negotiating a first mortgage of £75,000 to be secured on the property (*cf. J.*, 1921, 108 n).—(May 31.)

#### *British Dyestuffs Corporation.*

Capt. W. Benn asked the President of the Board of Trade whether the British Dyestuffs Corporation had refused to supply British export firms with synthetic indigo on the ground that the Corporation had its own agents in the Far East; and what steps the Government proposed to take in the matter. Sir P. Lloyd-Greame replied to the effect that the arrangements made by the British Dyestuffs Corporation with its agents in China preclude the sale of its products to merchants in this country when the goods are destined for export

to, and sale in, China. Merchants who supply the Chinese market can, however, obtain their supplies through the Corporation's agents in that country without disclosing information as to the ultimate purchasers. The Government is not responsible for the management of the British Dyestuffs Corporation and the Board of Trade has no power to dictate to firms the terms of their agency agreements.—(June 6.)

#### *The Jamaican Citrus Industry.*

Mr. Churchill, answering Mr. Hard, said that he had received suggestions from the Governor of Jamaica to the effect that the citrus-fruit industry of the Colony would be benefited if the Canadian Government would grant certain facilities in addition to those provided under the Canada-West Indies Agreement; correspondence was now proceeding with the Dominion Government on the subject.—(June 1.)

#### *Safeguarding of Industries Bill.*

During the debate on the financial resolutions, which form the basis of the Bill, Mr. Wise (Ilford) argued in favour of the 33½ per cent. import duty on fine chemicals. In his constituency there was a large chemical factory, established in 1797, which was feeling German competition so acutely that it had almost closed down and about three-fourths of its workers was unemployed. Mr. A. M. Samuel said that the world's production of optical glass did not amount to 50 tons, valued at about £50,000 at present, but that amount would suffice for the manufacture of optical instruments worth £30,000,000; the production of this indispensable commodity should not be allowed to pass out of our hands. Criticism was levelled against the quality of the arc-lamp carbons produced in this country, but Sir P. Lloyd-Greame said that they satisfied Admiralty requirements.

The debate on the second reading of the Bill was held on June 7 and 8, and the motion for the second reading was passed by 312 votes to 92.

### LEGAL INTELLIGENCE.

**ALLEGED INFRINGEMENT OF GERMAN SULPHUR-BLACK DYE PATENT.** *A.-G. für Anilin Fabrikation in Berlin v. Levinstein, Ltd.*

On May 9 the Court of Appeal, consisting of the Master of the Rolls and Lords Justices Warrington and Younger, dismissed an appeal by the A.-G. für Anilin Fabrikation in Berlin and the liquidator of the Mersey Chemical Works from a judgment given by Mr. Justice Sargant in February, 1914, dismissing the action brought by them against Levinstein, Ltd., for alleged infringement of a patent (1151/1900) for the manufacture of Sulphur Black dyes.

Counsel for appellants said that the patents taken out from 1873 onwards for processes of manufacturing dyes involving reaction between an alkali sulphide, sulphur, and an organic substance culminated in the Cassella patent of 1896, which, however, never came into use in this country. In these processes melting was necessary, and in 1900 appellants patented a process in which, although the reagents employed were the same as those specified in the Cassella patent, the melting was replaced by boiling. According to the appellants' specification, sodium sulphide and dinitrophenol were to be used, but in practice these were replaced by sodium dinitrophenolate. As the respondent company had not used dinitrophenol but

its sodium salt, it had been held that there was no infringement. Appellants, however, claimed that the respondents' process was a colourable imitation of theirs and that the two processes were chemically identical.

For the respondent company it was submitted that the Cassella process was not a melting process but a boiling process, and that there was no subject matter for the appellants' patent. If, however, the Judge was right in holding that there was sufficient invention in that patent to give it subject matter, then it followed that the use of the sodium salt in the Levinstein process gave it sufficient distinction from the appellants' process to make it impossible to allege infringement. All arguments to show that there was subject matter applied equally to show that there was no infringement.

In dismissing the appeal Lord Justice Warrington said that the defence of insufficiency of subject matter and insufficiency of description failed. The main issue was that of infringement, and the Court had concluded that the appellants' specification was confined to a process of boiling dinitrophenol. The use by Levinstein, Ltd., of sodium dinitrophenolate was a different process, and the holding of this salt with the solution mentioned in the appellants' specification would not be covered by their patent unless it could be proved that this process was common knowledge in 1900, the date of the patent. Appellants had not shown this, and their claim for damages for infringement failed.

### REPORTS.

**REPORT OF THE INDUSTRIAL ALCOHOL COMMITTEE, INDIA, 1920.** Pp. 62. (*Simla: Superintendent Government Central Press.*)

The Industrial Alcohol Committee was appointed to consider and report on the extent to which the existing excise regulations in India would require amendment in view of the possibility that the large-scale manufacture of industrial alcohol might be undertaken in the near future. The Committee found that the existing regulations, although adequate at present, would need alteration; that restrictions in production would have to be maintained even if those on sales, storage and transport were relaxed; that one possible obstacle to the manufacture of industrial alcohol is the seeming confusion of the rules concerning it; and another the restriction imposed by the Railway Board on the transport of alcohol in tank wagons which should be abolished.

The recommendations of the Committee include the following, among others:—Experiments should be made to determine if alcohol 55° overproof, denatured with 0.5 per cent. caoutchoucine, 1 per cent. pyridine, and a certain proportion of methyl violet, can be safely released from all excise restrictions except those regarding renaturation; and if such experiments prove successful, further tests should be undertaken to ascertain if spirit so denatured could be used advantageously in internal-combustion engines. If these further experiments were also satisfactory, or a better denaturant were discovered, a standard denaturant should be adopted for power alcohol throughout the whole country: such alcohol should be exempted both from excise duty (whether it be made in the country or be imported and then denatured) and from duty under the Motor Spirit Act. Power alcohol should be transported by rail on the same terms as dangerous petroleum; and special licences should be granted for the use of rectified or specially denatured spirit in industries.

An appendix to the report contains, in tabular

form, a list of industries in which alcohol is used, the form of alcohol which might be employed, the suggested place of denaturation, and suggested methods and formulae for industries requiring specially denatured spirit.

REPORT ON THE COMMERCIAL, INDUSTRIAL AND ECONOMIC SITUATION OF ITALY IN 1920. By SIR E. CAPEL CURE, Commercial Counsellor, and J. H. HENDERSON, Commercial Secretary to H.M. Embassy, Rome. Pp. 48. Department of Overseas Trade. London: H.M. Stationery Office, 1921. Price 1s.

The war left Italy in a disorganised and exhausted condition, but as natural resources and advantages, except possibly that of cheap labour, are still intact, eventual recovery may be anticipated; the chief obstacles thereto at the present time are labour unrest and depreciated currency. In 1919 industrial materials formed 52.5 per cent. of the imports, foodstuffs 32.2 per cent., and manufactured articles 15.3 per cent., these figures comparing with 57.3, 23.4, and 19.3 per cent., respectively, in 1913. The excess of imports over exports rose from 1134 million lire in 1913 to 11,328 millions in 1919, but there was a notable improvement during the first eight months of 1920. In 1919 the share of the United States in the foreign trade was 50 per cent. of the imports and 9 per cent. of the exports; that of Great Britain was 14 and 13 per cent., respectively. Germany, formerly first, now takes the sixth place with a volume of trade one-sixth that of the United States and one-fourth that of Great Britain. Germany supplies chiefly machinery, iron and steel, dyes and small hardware goods, and appears to be rapidly regaining its former ascendancy. There has been an enormous increase in the capitalisation of joint-stock companies since 1916; this still continues, and will undoubtedly be a grave source of danger when prices and profits decline; the net increases, in millions of lire, of chemical companies were 172.1 in 1918 and 133.2 in 1919, and of metallurgical companies, 725.5 and 214.2, respectively. Concomitantly, the values of industrial securities have seriously depreciated; in the chemical industries the index number fell from 100 in December, 1919, to 78.59 at the end of October, 1920, and that for the iron and steel industry from 102.49 to 51.10.

Agriculture showed a significant improvement in 1920, but as over 90 per cent. of the country is already cultivated, increased production will only be achieved by improved methods of cultivation. The annual production of citrus fruits, of which 52 per cent. is lemons, is about 800,000 metric tons, and that of essences and calcium citrate about one-tenth of the world's output. The sugar-beet, but recently introduced, is now cultivated on a large scale, and the national factories are producing nearly sufficient sugar to meet the home demand. Increased production of fertilisers and development of hydro-electric power are signs of the progress being made; but many years must elapse before the latter can have any marked effect on industrial productivity. A Government commission has been appointed to investigate Italian fuel resources, but it is doubtful if the results will justify the heavy expense, especially for boring, that will have to be incurred. Mineral production is probably much lower than the potential output, and the mineral wealth of the redeemed provinces is unlikely to affect the general position. During 1919 Venezia Tridentina and Venezia Giulia produced:—Copper ore, 26 metric tons; lead ore, 1082 t.; zinc ore, 20,149 t.; iron pyrites, 100 t.; mercury (metal), 237 t.; lignite, 93,300 t.; coal, 870 t.

## OFFICIAL TRADE INTELLIGENCE.

(From the Board of Trade Journal for May 26 and June 2.)

### OPENINGS FOR BRITISH TRADE.

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W. 1, from firms, agents or individuals who desire to represent U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number:—

Locality of firm or agent	Materials.	Reference number.
British West Indies	Drugs, bottles, chemicals for confectionery and aerated water trades .. ..	667
Canada .. ..	Vegetable colours for jam and confectionery, dyes for textiles, pigments .. ..	641
" .. ..	Pigments, dry colours, lithopone, litharge, zinc oxide, china clay .. ..	646
" .. ..	Medicated confectionery .. ..	668
" .. ..	Insulating varnish .. ..	671
" .. ..	Mining supplies .. ..	674
" .. ..	Chinaware .. ..	675
Egypt .. ..	Calcium carbide (tender for) .. ..	—
Italy .. ..	Metals .. ..	686
Spain .. ..	Timplate .. ..	657
" .. ..	Rubber .. ..	658
Smyrna .. ..	Sugar, paper, chemicals, carbide, petrol, pottery, glass, perfumery .. ..	660
South Africa .. ..	Paper .. ..	680
Netherlands East Indies .. ..	Chemicals .. ..	9744 F.W./P.H.
United States .. ..	Dry white lead .. ..	697
Argentina .. ..	Heavy and fine chemicals .. ..	664

MARKETS SOUGHT.—A company in eastern Canada about to manufacture milk powder wishes to arrange for sale in the U.K.

[Inquiries to the Canadian Government Trade Commissioner, 73, Basinghall Street, London, E.C. 2.]

### TARIFF. CUSTOMS. EXCISE.

*Australia.*—Recent customs decisions affect glucose, perfumed spirit, candles, matches, and paraffin wax.

*Belgium.*—The import of malt and the export of malt and matches are now permitted without licence.

*British West Africa.*—The import duty on spirits has been increased.

*Canada.*—It is proposed to amend, as from May 10, the import duties on cocoa, chocolate, coconuts, sugar, alcohol, spirits, and perfumes.

*Denmark.*—The export of potash and potash salts is allowed as from May 3.

*Ecuador.*—Sugar may now be exported subject to an export tax of 15 per cent. *ad valorem*.

*France.*—Until further notice the export is permitted of margarine, edible vegetable fats, bauxite, and iron ore.

*French West Africa.*—The import is prohibited of "trade spirits," thuyone, badian, benzoic aldehyde, salicylic ether, hyssop, and absinthe.

*Germany.*—Entry permits are required on entry into unoccupied Germany from occupied Germany of cocoa, cocoa husks, chocolate, artificial honey, refined sugar, margarine, compound lard, fertilisers, artificial sweetening matter, rubber tubing and tyres, asbestos wares, etc.

**Jamaica.**—The export of cocaine, opium, and certain explosives is prohibited to all destinations other than the British Empire.

**Latvia.**—Special export licences are no longer required for lime, gypsum, pottery, cardboard, cork, china, glassware, matches, soap, syrup, tar, turpentine, varnish, and window-glass.

**Mexico.**—The import of oleostearin is duty free.

**Netherlands.**—Export prohibitions have been withdrawn from ferruginous earth containing sulphur, and potassium salts.

**New Caledonia (French).**—The export duties on raw hides and skins and copra have been abolished, but minerals, whether in the form of ore or matte, pay 3½ per cent. *ad valorem*.

**Paraguay.**—The export duty on quebracho extract has been reduced to \$3 (gold) per metric ton.

**Peru.**—Vanadium ores with less than 10 per cent. of vanadic acid may be exported duty free.

**Spain.**—The full text of the new provisional Customs Tariff is issued as a supplement to the issue of May 26. The tariff is to remain in force pending a further revision of a more permanent character and is operative as from May 21.

All classes of goods are affected, and in most cases the duties are increased. Among the articles on which the duties have been reduced are china-ware, perfumery, spirits, and rubber wares. Additional surtaxes are levied on petroleum, coconut oil, palm oil, indigo, manilla hemp, agave, jute, raw hides and skins. Export duties are payable on cork, bones, galena, litharge, lead ores, argentiferous lead, iron ore, copper ore and copper matte. Sheet, plate, and crystal glass when imported coloured, bevelled or engraved pays in respect of each such operation a surtax of 10 per cent. of the duty applicable. Bimetal sheets are classed under the heading of the more highly taxed metal. Tanned or impregnated timber pays the duty applicable plus a surtax of 10 per cent.

**Sweden.**—Export prohibitions have been withdrawn from mineral and lubricating oils.

It is proposed to increase the import duties on many classes of goods. The schedule of articles affected is set out in the issue of June 2.

**United States.**—The Emergency Tariff Bill became effective on May 28. For a period of 3 months the admission is prohibited of sodium nitrite, dyes, crudes or intermediates, products derived directly or indirectly from coal tar, and synthetic organic drugs or chemicals, unless a satisfactory substitute therefor is unobtainable in the United States.

## GOVERNMENT ORDERS AND NOTICES.

**GERMAN REPARATION (RECOVERY) ACT.**—An Order has been issued by the Board of Trade under the above Act exempting from its provisions any article proved not to have been first consigned from Germany to the United Kingdom. Goods are regarded as consigned from the country where they were last owned, and from which they were procured by the importer. The Order No. 5 has been cancelled.

**LEATHER MEASUREMENT REGULATIONS, 1921.**—In view of the increasing use of leather-measuring instruments in the leather trade, regulations have been made by the Board of Trade under the Weights and Measures (Leather Measurement) Act and will come into force on July 1. After that date every leather-measuring instrument of an approved pattern must, if used for trade, be verified and stamped by an Inspector of Weights and Measures within a period of twelve months.

## TRADE NOTES.

### BRITISH.

**British Guiana in 1919.**—Owing to scarcity of labour, bad weather, and shortage of nitrogenous fertilisers, the sugar crop amounted to only 86,971 tons, of which 83,140 t. was exported, mainly to the United Kingdom. The acreage under cacao remained at about 2000 acres, but the planting of Para rubber decreased. Para rubber grows well on suitable lands in the colony and gives good yields, but is susceptible to leaf disease. Balata bleeding was continued and 627·3 t. was exported. Coconuts are now planted over 27,400 acres, an area which could be greatly increased; 4,693,000 nuts, 68 tons of copra, and 9120 galls. of coconut oil were exported. Limes are cultivated over about 1350 acres, and the erection of factories for the production of concentrated juice and citrate of lime has given the industry a much-desired impetus; the exports were 389·5 cwt. citrate of lime, 7356 galls. of raw and 2882 galls. of concentrated lime juice, and 326 galls. of oil of limes. During the year 16,216 oz. gold and 16,706½ carats diamonds were produced, and eight licences were issued for mineral oil exploration in the north-western and Pomeroon districts, but no definite results were recorded. The principal exports include sugar, rum, molasses, molascuit, rice, and citrate of lime. Leather, cacao and matches are manufactured, and there is a large ice factory. The value of the exports was £4,240,832, an increase of 20·21 per cent. on 1918 and imports were valued at £3,590,812. The United Kingdom takes first place with 34·13 per cent. of the total trade of the colony, followed by Canada and the United States with 30·54 and 17½ per cent., respectively.—(*Col. Rep.—Ann., N. 1068, Mar., 1921.*)

### FOREIGN.

**Imports of Chemicals into the Dutch East Indies.**—H.M. Commercial Agent at Batavia, Netherlands East Indies, has supplied the Department of Overseas Trade with the following statistics relating to imports of chemicals into the Netherlands East Indies during 1919.

The following statement shows the quantity and value of the various chemicals imported into the Dutch East Indies during 1919:—

	Quantity. Metric tons.	Value. £
Alum .. ..	1,620 ..	16,190
Ammonia .. ..	33 ..	9,080
Acetic acid .. ..	221 ..	31,310
Calcium carbide .. ..	767 ..	32,830
Caustic soda .. ..	2,869 ..	99,000
Sodium nitrate .. ..	84 ..	2,670
Sodium sulphate .. ..	1,791 ..	35,440
Copper sulphate .. ..	46 ..	2,080
Sulphur .. ..	3,298 ..	43,910
Sulphur pyrites .. ..	9,160 ..	48,180
Sulphuric acid:—		
Commercial .. ..	914 ..	17,460
Refined .. ..	38 ..	1,210

Imports into Java and Madura were valued at 3,745,972 fl. (£331,500) and those into the outlying possessions at 2,431,373 fl. (£215,000), making a total of 6,177,435 fl. (£546,500) for the whole of the Netherlands East Indies. Direct imports from the United Kingdom totalled 1,165,631 fl. (£103,150) or approximately 18·5 per cent., but the imports from Singapore amounted to nearly 15 per cent. of the total, and it is probable that a considerable portion of this represents chemicals in transit from the United Kingdom. Japan supplied 30 per cent. of the total chemicals imported, a large item being sulphur pyrites (529,866 fl.) imported by the Bataafsche Petroleum Mij. for its sulphuric-acid plant at Balikpapan in Borneo. The share of the United States was approximately 25 per cent., one-third of this total consisting of caustic soda and one-third of chemicals not enumerated.

Owing to the advance of scientific agriculture, the development of new plantations and the opening up of new industries, the chemical trade of the Netherlands East Indies is of growing importance. Competition for this market from Japan and the United States of America has to be considered, and Germany is already sending out agents in an endeavour to regain the trade lost during the war. United Kingdom firms are advised to send out representatives to canvass the market and be in a position to quote c.i.f. prices for immediate or forward delivery. As an alternative, much might be done by forwarding catalogues to H.M. Commercial Agent at Batavia for delivery to chemical importers, or by advertising in the East Indian Press. Catalogues and advertisements, however, would be of little value unless prices were quoted. It should be possible not only to maintain United Kingdom trade in chemicals with the Netherlands East Indies, but also to increase it, as there is every indication that the Japanese are losing their hold on the market, which is gradually favouring the United States and Germany. The names of importers of chemicals in the Netherlands East Indies and of United Kingdom advertising contractors may be obtained on application to the Department of Overseas Trade (Reference No. 9744.F.W./P.N.).

**The Dutch Soap Trade in 1920.**—Although in normal times soap is manufactured and exported from Holland in large quantities, there is a considerable import trade, particularly in toilet soaps. During 1920 imports declined and the export trade recommenced. Imports of various grades of soaps, together with the chief countries of origin, are given below:—

	Metric tons.	1919.		Metric tons.	1920.	
		Per cent.			Per cent.	
cented, transparent, and liquid	604	U.K. 84, U.S. 59, Belg. 2		621	U.K. 43, Fr. 12, Ger. 11.5, Belg. 11, U.S. 10	
Hard, unscented, and powder	2,348	U.K. 95, U.S. 5		691	U.K. 50, U.S. 50	
Soft .. ..	81	—		70	—	
Textile .. ..	12	—		0	—	

The figures for 1919 include the transit trade, but those for 1920 do not; the volume of this trade is about 20 per cent. of the figures given for 1920. Exports in 1919 and 1920 were as follows:—

	1919.		1920.	
	Metric tons.		Metric tons.	
Scented, etc. .. ..	216		432	
Hard, unscented .. ..	2,524		3,521	
Soft .. ..	130		229	
Textile .. ..	—		23	

The percentage representing transit trade should be added to the returns for 1920. About 75 per cent. of the exports of toilet soap in 1920 went to the Dutch East Indies, and the balance to Belgium and Germany; and of the hard soap exported, 40 per cent. went to Germany, and the rest mainly to Poland, Scandinavia, and the Dutch East Indies, in the order named.—(U.S. Com. Rep., Mar. 19, 1921.)

**Foreign Company News.—Brazil.**—It is reported that a large cement works is being constructed at Belem, Para, for the Companhia Para-Ceara, which intends to produce cheap cement for the reclamation work to be carried out in the State of Ceara.—(U.S. Com. Rep., Apr. 13, 1921.)

**Germany.**—The Oberschlesischen Kokswerke und Chemische Fabriken A.G. has made a profit of 16,890,855 mk.; a dividend of 17 per cent. has been declared and 886,419 mk. carried forward (capital 6,800,000 mk.).

The net profit for 1920 of the Rütgerswerke A.G. in Berlin was 4,802,661 mk.; 3.8 million mk. has been written off and 3 million mk. carried to maintenance account. The dividend is 15 per cent. (12½ per cent.) and bonus 150 mk. (capital 37,500,000 mk.).

With the co-operation of the Chemische Fabrik auf Aktien (vorm. E. Schering) in Berlin, the Richard Jahr dry-plate factory has been converted into a limited liability company with a capital of 2.4 million mk.—(Chem. Ind., Apr. 18, 25, 1921.)

**Poland.**—The Polish Chemical Works in Cracow (capital 50 million mk.) has begun to erect a factory at Bogumin (Oderberg) for the manufacture of saccharin and other chemical products.

A company to manufacture explosives has been founded under the name of the "Nitrat" Chemical Works (capital 300 million mk.) by the A.-G. Pocisk and the Tomaszower Artificial-Silk Works.—(Chem. Ind., Apr. 18, 1921.)

**Sweden.**—The proposal to manufacture pharmaceutical products at a central laboratory connected with the A.-B. Svenska Färgämnesindustri in Södertälje (cf. J., 1921, 33 n) has been abandoned, and it is now proposed that such a laboratory be attached to the Svenska Medicinalväxtförceningen, a company which owns a large works at Ljungby. If this takes place the sale of pharmaceutical products in Sweden will become centralised.—(Chem. Ind., Apr. 18, 1921.)

**United States.**—The net profit of the Allied Chemical and Dye Corporation in 1920 was \$16,179,939. A dividend at the rate of \$6.33 per share has been declared on the 2,143,455 shares of common stock.—(Oil, Paint and Drug. Rep., May 16, 1921.)

The Manito Chemical Co. has recently completed a plant at Manito, Illinois, which is stated to be one of the largest plants for preparing natural humus in the world. It is situated on a bed containing over one million tons of humus; the product is black, completely decomposed, and contains 12–14 per cent. water and 3–5 per cent. ammonia.—(Amer. Fertiliser, Mar. 26, 1921.)

## COMPANY NEWS.

**BRITISH CYANIDES Co., LTD.**—The directors announce that owing to the industrial situation they are unable to recommend the payment of a dividend on the ordinary shares. The company's works are closed owing to lack of coal, but work on the plant for the fixation of atmospheric nitrogen is being continued and nearing completion.

**JURGENSEN, LTD.**—The profit for the year 1920 was £377,467, as against £222,040 in 1919. Including the amount brought forward the available balance is £146,280, from which £251,206 has been applied to the writing down of stocks to market value, an amount equivalent to a 10 per cent. dividend on the ordinary shares, upon which no dividend is payable. Satisfactory progress is reported at the company's factory at Purfleet.

**BRITISH COTTONSEED PRODUCTS, LTD.**—At the first annual meeting held on May 23, the chairman stated that the company now owned the whole of the world's rights for the seed-defibrating machine (cf. J., 1918, 112 r, 1919, 461r), with the exception of the United States; and that it was hoped to launch the Indian company during the present year. The erection of plant and machinery in the works at Chatham had been delayed, and in view of the high price of local electricity supply, the company had decided to erect a power-plant of its own.

**CAPE EXPLOSIVES WORKS, LTD.** has successfully issued £1,000,000 of 7½ per cent. first mortgage debenture stock at 95 per cent., the repayment of which is guaranteed by De Beers Consolidated Mines, Ltd. The company's works are situated at Somerset West, Cape Province, S. Africa, and its manufactures include explosives, fertilisers, sheep

and cattle dips and other chemical products. A plant has recently been completed which is able to supply the whole of the Union's requirements in detonators, and the fertiliser plant is capable of producing 70,000 tons per annum. The issued share capital consists of 500,000 ordinary shares of £1 each, all of which are held by the De Beers Company, and the profits, before allowing for depreciation and taxation, have lately been at the rate of about £146,700 per annum, but the fertiliser plant has only recently come into operation.

**NATIONAL DYES, LTD.**—The first annual report and accounts covering the period December 15, 1919, to December 31, 1920, were presented to the shareholders on June 6. The report shows that 786,703 lb. of colours and 306,848 lb. of intermediates were manufactured at the company's works during that period, which is considered satisfactory, especially having regard to the fact that the trade depression necessitated the complete closing down of the factories before the end of the year. The accounts show a gross profit, after making suitable provision for decline in market value of certain raw materials, of £62,098, and after writing off £13,034 depreciation on plant, tools and machinery, £17,080 debenture and bank interest, also bad debts, etc., a balance of £6,610 is carried forward to the debit of profit and loss account.

**THE BRITISH OIL AND CAKE MILLS, LTD.**—Addressing the 23rd ordinary general meeting on May 31, Mr. J. W. Pearson, chairman, said that the coal strike had entailed no stoppage of the company's operations, as practically all the boiler plants had been converted to use oil fuel, approximately 75 per cent. of the entire output being obtained by this means; and that certain of the refinery refuse could be profitably burnt in the converted plant. Except at such expensive centres as London, he was doubtful of the superior economy of oil fuel; further north, where coal could be supplied at less than half the price in London, the advantage was entirely in favour of coal. The profit and loss account for the year 1920 shows a balance of £414,464, which with £22,010 brought in allows of a total dividend of 15 per cent. on the ordinary shares, compared with 25 per cent. for 1919, when the ordinary share capital was one-half of what it is now (£2,500,000). Stocks at the end of the year were exactly the same as at December 31, 1919, but the value was assessed at one-half.

**BRITISH CELLULOSE AND CHEMICAL MANUFACTURING Co., Ltd.**—A progress report states that the board has adopted the recommendations made by Mr. A. Chamberlain (a director) in December last which proposed an immediate and drastic restriction of programme, including the cancellation or postponement of the delivery of much plant. The present position is that plant to produce 1 ton of silk per day is nearing completion; the daily output is about 1000 lb., and it is expected to increase this to 1 ton by the end of August. To make provision for an output greater than 1 ton per day, the sum of £278,000 has been borrowed and secured by a debenture. Any further increase in output capacity will require further cash. The directors are quite satisfied with the quality of the silk, which dyes well and gives results in many respects superior to those obtained with other artificial silks. The Post Office has approved the use of the silk for insulating purposes. Owing to the importations of carbide and other chemicals from the Continent, the carbide and other chemical departments were closed down early in the year, but it is hoped to reopen them if the Safeguarding of Industries Bill becomes law and these branches of the company's work are included within its scope. The production of non-inflammable cellinoid is making good progress.

## REVIEWS.

- (1) **ECONOMIC MINERALOGY.** By THOMAS CROOK. Pp. xi+492. (London: Longmans, Green and Co. 1921.) Price 25s. net.
- (2) **GEOLOGY OF THE NON-METALLIC MINERAL DEPOSITS OTHER THAN SILICATES. VOL. I. PRINCIPLES OF SALT DEPOSITION.** By A. W. GRABAU. Pp. xvi+435. (New York and London: McGraw-Hill Book Co., Inc. 1920.) Price 30s. net.

No single treatise can essay to cope with the facts and principles of so complex a subject as economic mineralogy or geology in a manner that will satisfy the needs of specialists; they have to turn to the books dealing with their specific subjects, it may be gold, zinc, oil, clay, and so on. The student who requires something more than a catalogue and less than a monograph is fairly well provided for by DeLaunay, Stutzer, Dammer and Tietze, Krusch and Beyschlag, and a goodly array of works on the genesis and distribution of ore minerals. But, judging by the recent output of such books, there is evidently a demand for a condensed practical guide to the study of useful minerals, and no book yet published in English has more successfully achieved this object or contains more information for its size than does Mr. Crook's mineralogy. Nothing, however, could illustrate more clearly than the two books under review the wide divergence that is possible in the treatment of the subject; the first is in effect a compact encyclopædia, the second is an attempt to treat one broad aspect of the subject philosophically, but with an ulterior economic motive.

(1) Mr. Crook begins with a very brief account of crystals and the general physical characters of minerals. He next explains the practical elements of crystal optics and this he does with unusual clarity. The simple chemical and blowpipe tests and laboratory methods of separation and concentration are adequately dealt with, though a little more might have been said about the employment of vertical and oblique illumination with white and monochromatic light in the study of opaque minerals in polished surfaces. Still, all this introductory matter bears the obvious mark of the experienced manipulator. The chapter on the geology of economic mineral deposits was perhaps necessary, it is of course just geology, and it was naturally impossible, in the allotted space, to give it proper treatment. In the descriptive portion of the volume the economic minerals are arranged in three groups: (1) ore minerals, (2) gem minerals, (3) miscellaneous minerals. Within each group the arrangement is alphabetical. By "ore mineral" the author means "a metalliferous mineral which owes its economic value to the fact that it is smelted to obtain a metal." This is not true, and if it were it would be misleading, for some of the metals included in this group are in practice more often extracted by electrolytic or purely chemical processes. Amongst the gem minerals are included many of little value or that are rarely employed. The miscellaneous minerals are described partly with reference to their uses, e.g., abrasives, building stones, cement materials, refractories; partly in accordance with their chemical classification, e.g., carbonates, sulphates, silicates, borates, and partly under common mineral names, e.g., asbestos, mica, clays. This section is full of information though in an abbreviated form; it is fairly well up-to-date but, as would be expected, there is some inequality in treatment; the remarks under building stones and cement materials, for example, do not take one very far; lime is mentioned under fertilisers, but there seems to be no reference to



chemical lime. Flint is almost passed by and it is not indexed. Useful determinative tables come at the end of the book. Cross references have been carefully made and the index is satisfactory, but there are no references to original authorities. Most of the illustrations are good and new.

(2) Any work from the pen of Dr. Grabau is deserving of attention and this will doubtless be true of the one now under consideration. In the author's own words, it is "essentially a treatise on applied stratigraphy." The present volume treats of the principles of salt deposition, as deduced from the study of such deposits now forming or which have but recently been formed. These principles are to be applied in a second volume to the elucidation of the older deposits. The term salt is used with a very wide connotation. The composition of natural saline waters is reviewed together with the laws governing the deposition of salts in nature. The investigations of Van't Hoff, Walther and others and the experiments of Usiglio are discussed. Different types of deposit are examined—sea-margin deposits, formations in lakes and cut-offs, cyclic or wind-borne salts, connate salts and those derived from pre-formed rock bodies. A chapter is devoted to the concentration of salts of different kinds by the vital activities of animals and plants, another to the deposits from mineral springs, fumaroles, and circulating ground-waters. The "meta salts," that is, those formed *in situ* by metasomatic processes, are disposed of rather briefly. The interesting and difficult subject of deformation in salt bodies has a chapter to itself.

As regards the principles of salt deposition, the author rather frequently avoids giving a definite lead, preferring to quote variant hypotheses and leave the choice to the reader.

Though this is only the first instalment of a rather ambitious scheme, and the final judgment upon it must be deferred, it will be found a very handy introduction to the study of salt deposits and the distribution of the younger salt formations. The numerous outline maps and references to literature enhance its value in this respect.

J. ALLEN HOWE.

TRAITE DE LA CONSERVATION ET DE L'AMELIORATION DES BOIS. By M. DE KEGHEL. *Encyclopédie Industrielle*. Pp. 360. (Paris: J.-B. Baillière et Fils. 1921.) Price 10 francs (bound 15 fr.).

There have been very few works attempting to handle this important subject as a whole, although many papers or pamphlets have been written. In the present case the author has condensed into 360 pages of a handbook an enormous amount of information which could only be dealt with in many volumes.

The matter is divided under 18 headings or chapters. The first is historical and critical, and leads up to the final paragraph which states that a rational and complete impregnation of wood is only possible by the application of a system in harmony with a knowledge of its anatomical structure and histology. These subjects are dealt with in Chapter II which shortly goes into the cause of the circulation of the sap and the course it follows. After a few remarks on the cellular and vascular tissue and the chemical composition of wood, the author proceeds to show the difficulties in the way of thorough impregnation of all parts; and, in Chapter III, how these may be overcome. He proposes to drive out the sap by a longitudinal injection of a suitable fluid. Chapter IV describes the author's plant which provides for the immersion of the log in the fluid under the same pressure as is applied to one end, whilst the other end is open to atmosphere. This ingenious arrangement is to

prevent the hursting of the wood under the high pressure necessary. Chapter V is on the selection and felling of the trees and the preparation of the logs for the special treatment, whilst Chapter VI, on the artificial ageing or senilisation of wood, is of very great interest. The changes brought about by natural and artificial ageing are explained and the reasons given for the failure of wood seasoned by the ordinary artificial methods. It describes several unsatisfactory processes which pretend to overcome these disabilities. All this leads up to the author's process of the removal of the sap and soluble constituents by forcing through the logs a weak aqueous solution of sulphuric acid of ammonia, etc., followed, after draining, by treatment with acetone. Subsequent drying in special plant results in the recovery of a very large proportion of the acetone, and the preparation of a wood which will not warp, swell or crack. The author shows that it is possible by washing to remove the hygroscopic soluble and putrescible matter from the woody structure, and that then the drying is rapid. He gives figures showing the differences in weight and time of drying of treated and untreated wood.

Chapters VII to XIII deal with fireproofing historically, chemically, and industrially. Failures are explained and the author's investigations leading up to his zinc-ammonio-phosphate solution are described.

Chapter XIV is a very long one handling the subject of wood preservation from the industrial point of view. It opens with about four pages on the causes of destruction, and then goes on to describe and criticise many of the processes which have been proposed or used for preservation. This naturally leads to the rational methods invented by the author, as exemplified by the longitudinal injection of an aqueous solution of agar-agar, formaldehyde, and ferrocyanide of potassium for railway sleepers, and a solution which will deposit sulphate of lime in the case of wood intended for paving.

Whilst the author's reasons for adopting such processes appear to be sound and to be supported by experimental data, it cannot be overlooked that he strongly condemns creosote as a preservative, making use of examples of inefficient application, and arguing that its viscosity and other disabilities make it impossible to cause it to penetrate the wood. He apparently ignores the fact that sleepers, telegraph poles, piling, and many other creosoted structural timbers have lasted much beyond their economical or mechanical life. Large fencing posts erected in 1854 are still sound. His comparative experiments with beech sleepers against white ant must be discounted. If the amount of time and care given to his agar process had been expended on the control sleepers which were creosoted, they would not have suffered.

Chapter XV is upon the colouring of wood, either to imitate others or to produce "façy" effects. The author has given a great deal of thought to this subject, and his reasons for failures and proposals for obtaining successful results are supported by experiments. He introduces the theory of colloids to explain the unequal absorption of colouring matters, and how wood may be mordanted to render all its constituents of the opposite electric sign to that of the colouring matter. He explains how, in any haphazard mixing of colouring matters, the different viscosities of the constituents would result in selective separation in the capillaries; but he states that a complex of definite composition, more stable than either of the constituents, may be formed by mixing certain positive or basic colours with negative or acid colours.

There is a chapter on plywood, compressed wood, and bent wood, and finally, one on the installation of plants suitable for carrying out the author's ideas.

The diagrams and reproductions of actual plant are badly printed; otherwise the perusal of the book has been one of pleasure and instruction. It is to be regretted that the author has said little or nothing about the great plants of England and America, or of the large amount of work of investigation carried out by the American Wood Preservers' Association and others. This may be accounted for by the fact that most of these plants use the creosote process.

HUBERT FERGUSSON.

**RED LEAD AND HOW TO USE IT IN PAINT.** By A. H. SABIN. *Third edition, rewritten and enlarged.* Pp. xi+139. (New York: J. Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1920.) Price 11s. 6d. net.

This work is mainly intended for the use of constructional engineers, architects, and others who have to deal with the protection of iron from rust. The author lays great stress upon the utility of red lead for this purpose, and he rightly points out the importance of the fineness of division of the red-lead particles, so that a most homogeneous paint-coating may be obtained. A great deal of importance is attached to the amount of litharge present, and the author advises the use of a pure red lead because it is found that on keeping as a mixed paint the litharge causes "livering," or if much be present the paint may even set hard; in any case such paints cannot be spread evenly. The exact reasons why pure red lead ( $Pb_3O_4$ ) should be superior to other pigments as a protective coating for iron are not made clear; the author, as stated, insists upon its fineness, but does not say why any equally fine inert pigment should not do as well. Although the presence of much litharge is condemned, some may be added, if desired, under special circumstances to a 98 per cent. red lead paint. The reviewer is of the opinion that insufficient importance has been attached to the character of the oil binder used for thinning (*cf.* Ingle and Woodmansey, J., 1919, 101t); but the mention of the sand blast as a means of cleaning rusted surfaces either for painting or repainting is a welcome suggestion; it has been used in America for some time, but does not seem to have found sufficient application in Europe. The book is embellished with numerous photographs of bridges, steamers, etc., which seem to serve little useful purpose, but as the volume is written in a popular style, it is suitable for the people for whom it is intended.

HARRY INGLE.

## PUBLICATIONS RECEIVED.

**THE CASE-HARDENING OF STEEL.** By H. BREARLEY. *Second edition.* Pp. 207. (London: Longmans, Green and Co. 1921.) Price 16s.

**INTERNATIONAL HANDBOOK OF THE WORLD'S CHEMICAL INDUSTRY AND TRADE, 1913-1914-1919-1920.** By DR. W. A. DYES. Volume I. Pp. 1-752. Edition E. (Wittenberg near Halle: Hopf'sche Verlagsbuchdruckerei Gebr. Jenne, G.m.b.H. 1921.)

**FESTSCHRIFT DER KAISER WILHELM GESELLSCHAFT ZUR FÖRDERUNG DER WISSENSCHAFTEN ZU IHREM ZEHNJÄHRIGEN JUBILÄUM.** Pp. 282. (Berlin: Julius Springer. 1921.) Price: paper 100 mk., bound 130 mk.

**TANNING MATERIALS.** By A. HARVEY. Pp. vi.+182. (London: Crosby Lockwood and Son. 1921.) Price 15s.

**THE CHEMICAL EFFECTS OF ALPHA PARTICLES AND ELECTRONS.** By S. C. LIND. *American Chemical Society. Monograph Series.* Pp. 182. (New York: The Chemical Catalog Co., Inc. 1921.)

**PERFUMES, ESSENTIAL OILS, AND FRUIT ESSENCES.** By GEOFFREY MARTIN. *Manuals of Chemical Technology, edited by Geoffrey Martin.* Vol. X. Pp. vii.+138. (London: Crosby Lockwood and Son. 1921.) Price 12s. 6d.

**FACTORY CHEMISTRY.** By W. H. HAWKES. Pp. 59. (London: Longmans, Green and Co. 1921.) Price 4s. 6d.

**PNEUMATIC CONVEYING.** By E. G. PHILLIPS. *Pitman's Technical Primer Series.* Pp. 108. (London: Sir Isaac Pitman and Sons, Ltd. 1921.) Price 2s. 6d.

**REVIEW OF AGRICULTURAL OPERATIONS IN INDIA, 1919-20.** Pp. 140. (Calcutta: Superintendent Government Printing, India. 1921.) Price, Rs. 1-4.

**AUSKUNFTSBUCH FÜR DIE CHEMISCHE INDUSTRIE.** By H. BLÜCHER. *Eleventh edition, revised and enlarged.* Part I. Pp. 797. Part II. Pp. 803-1590, with appendices. (Berlin and Leipzig: Vereinigung Wissenschaftlicher Verleger, H. de Gruyter and Co. 1921.) Price, bound, 35s. each part.

**PUBLICATIONS OF THE UNITED STATES BUREAU OF MINES. Department of the Interior. (Washington: Government Printing Office. 1920.):—**  
**STRUCTURE IN PALEOZOIC BITUMINOUS COALS.** By R. THIESSEN. *Bulletin 117.* Price 80 cents.  
**STATE MINING LAWS ON THE USE OF ELECTRICITY IN AND ABOUT COAL MINES.** By L. C. LISLEY. *Technical Paper 271.* Price 10 cents.  
**PRELIMINARY INVESTIGATIONS OF STORAGE BATTERY LOCOMOTIVES.** By L. C. LISLEY and H. B. BRUNOT. *Technical Paper 264.* Price 10 cents.

**STENCHES FOR DETECTING LEAKAGE OF BLUE WATER GAS AND NATURAL GAS.** By S. H. KATI and V. C. ALLISON. *Technical Paper 267.* Price 5 cents.

**CAUSES AND PREVENTION OF FIRES AND EXPLOSIONS IN BITUMINOUS COAL MINES.** By E. STEIDLE. *Miners' Circular 21.* Price 20 cents.

**TENTH ANNUAL REPORT BY THE DIRECTOR OF THE BUREAU OF MINES FOR THE YEAR ENDED JUNE 30, 1920.** Price 15 cents.

**PUBLICATIONS OF THE CANADIAN DEPARTMENT OF MINES. Mines Branch. (Ottawa: Thomas Mulvey. 1921.):—**

**THE PRODUCTION OF COPPER, GOLD, LEAD, NICKEL, SILVER, ZINC, AND OTHER METALS IN CANADA IN 1919.** (No. 547.)

**THE PRODUCTION OF COAL AND COKE IN CANADA IN 1919.** By J. McLEISH. (No. 548.)

**BULLETINS OF INDIAN INDUSTRIES AND LABOUR. (Calcutta: Superintendent Government Printing, India. 1921.):—**

**No. 1. PROCEEDINGS OF THE FIRST CONFERENCE OF DIRECTORS OF INDUSTRIES.** Price 1 rupee.

**No. 2. PROCEEDINGS OF THE SECOND CONFERENCE OF DIRECTORS OF INDUSTRIES.** Price 12 annas.

**PUBLICATIONS OF THE UNITED STATES GEOLOGICAL SURVEY. (Washington: Government Printing Office. 1921.):—**

**COKE IN 1918.** By C. E. LESHER and F. G. TRYON.

**GOLD, SILVER, COPPER, LEAD, AND ZINC IN CALIFORNIA AND OREGON IN 1919.** By C. G. YALE.

**ZINC IN 1918.** By C. E. SIEBENTHAL.

# INCOME AND EXPENDITURE ACCOUNT FOR YEAR ENDED 31st DECEMBER, 1920.

EXPENDITURE.		INCOME.	
Corresponding figures for 1919.			
£ s. d.	£ s. d.	£ s. d.	£ s. d.
To Journal—Editorial and Sundry Expenses—		By Entrance Fees .....	960 0 0
Editor's Salary .....	750 0 0	By Contributions .....	1509 0 0
— Expenses .....	43 3 5	By Journal .....	1509 0 0
		By Great Eastern Railway Co. Consolidated 4% Preference Stock .....	875 4 0
		By South Eastern Railway Consolidated 4% Preference Stock .....	400 9 0
		By Metropolitan Railway Consolidated 4% Preference Stock .....	510 0 0
		By London & North Western Railway Joint Station 5% Stock .....	93 0 0
		By Great Western Railway Co. 5% Consolidated Stock .....	1008 5 0
		By Great Northern Railway Co. 3% Debenture Stock .....	400 0 0
		By Nottingham and Grantham Railway Co. 4% Consolidated Stock .....	231 5 0
		By India 3 1/4% Stock .....	394 16 0
		By India 3% Stock .....	396 7 0
		By War Loan 3 1/4% Stock .....	584 10 0
		By War Loan 5% 1920-21 .....	1494 0 0
		By Office Furniture .....	579 4 0
		By Loss Depreciation .....	100 0 0
			£19,331 0 10
	£19,331 0 10		

To the Members of the Society of Chemical Industry.  
 That the above Balance Sheet, dated 31st December, 1920, with the Books and Vouchers of the Society, and have verified the Investments, and we are of opinion that such Balance Sheet correctly sets forth the financial position of the Society at 31st December, 1920.

S. Frederick's Place, Old Jewry, London, E.C. 2.  
 11th June, 1921.  
 PRICE, WATERHOUSE & CO.,  
 Chartered Accountants.



## THE RELATIVE VALUE OF THE PROTEINS IN NUTRITION.

R. H. A. PLIMMER.

Complete hydrolysis of the protein to its constituent 18 or 20 amino-acids occurs during digestion in animals; the amino-acids circulate in the blood and reach the various organs, which build up new tissue from the units. Animals have been maintained on a diet containing as its protein content a mixture of pure amino-acids in suitable proportions. Biologically, the proteins must therefore be regarded as mixtures of the various amino-acids, digestion and absorption as a re-shuffling of the units. The amino-acids are not convertible into one another, nor capable of being synthesised by the animal organism, with the exception of glycine, which, under certain conditions, can be formed in the body. The different proteins have different compositions, thus, for example, casein contains 13 per cent. of glutamic acid and gliadin 40 per cent. Some proteins are complete, i.e., contain all the amino-acids, others are incomplete and lack certain units. It can thus hardly be expected that proteins should have the same value in nutrition. Some of the amino-acids may be indispensable, others not so essential. There are two main problems to study in nutrition: the formation of new tissue, as in the growth of young animals, and the maintenance of tissue, which undergoes so-called wear and tear, in adult animals. The problem is ultimately to find out the function of each amino-acid in growth and maintenance.

### *The effect of the absence of certain amino-acids.*

The most certain way of studying the problem is to feed animals upon known mixtures of amino-acids, but the practical difficulties are far too great. The amino-acids are not easily prepared, and it is almost impossible to obtain sufficient of each of them to feed an animal, even a mouse, for any length of time. Two other ways are possible:—(a) To feed incomplete proteins and add the missing unit or units; (b) to feed completely hydrolysed proteins, i.e., a mixture of amino-acids from which one or more units have been removed by chemical means.

The first experiment of this kind was made by Willcock and Hopkins in 1906. Zein was chosen as an incomplete protein and fed to mice: in one set one, in another set with the addition of 2 per cent. of its amount of tryptophan. Failure occurred in both sets of animals, but not so rapidly in those with zein and tryptophan. Young mice with zein alone died generally in 16 days; with zein and tryptophan in 30 days. Adult mice without tryptophan lived 27 days, with tryptophan 49 days. The survival period was thus appreciably lengthened by the presence of tryptophan. The failure to thrive was most probably due to the absence of other units from the zein. Ackroyd and Hopkins repeated the experiment in 1916 by the second method of experimenting which offers better conditions. The animals were fed upon a mixture of the amino-acids from casein. This mixture does not contain tryptophan, since it is destroyed by acid hydrolysis. In the first period tryptophan was added; on the 12th day it was omitted and introduced once more on the 35th day. The animals continued their growth during the first period, declined in weight during the second period, grew again in the third period when the tryptophan was present.

Oshorne and Mendel in America have made numerous experiments with various pure isolated proteins of known amino-acid composition. Their

most important results in this connexion were with wheat gliadin; this is a complete protein, but has very little lysine. Adult rats were maintained for long periods on this protein—as long as 500 days—but young rats, though they lived for long periods, failed to grow. The authors therefore regarded lysine as essential for growth. Later, they showed that if they added lysine at definite intervals to the food containing gliadin as sole protein, growth took place with the lysine, but not without it. The minimum amount of lysine necessary to produce normal growth was found to be between 2 and 3 per cent. of the amount of protein in the diet.

The value of lysine for growth was shown in a more practical way by Buckner, Nollau and Kastle. They fed chickens on a poultry farm on diets of high and low lysine content. Their figures and photographs definitely showed more rapid growth on the mixture of high lysine content.

The two other hexone bases—arginine and histidine—are essential units of proteins. As shown by Ackroyd and Hopkins, their removal from the hydrolysed casein mixture leads to loss of weight of the animals; if only one is absent from the food the rate of growth is lessened, so that it appears as if these two units were inter-related. The structural formulae of these compounds suggest a possibility of the conversion of the one into the other by the wonderful mechanisms of the animal body. These two units were found to give an increase of allantoin in the urine of the animals; thus we know that the purine ring can be synthesised from them. This synthesis by animals has long been suspected, as young birds and animals produce purines on a diet with an almost complete absence of purine compounds.

It is more difficult to arrive at the function of the amino-acids containing aromatic nuclei. Some proteins lack tyrosine, but all contain phenylalanine, which is very difficult to remove from a mixture. Phenylalanine and tyrosine both give rise to homogentisic acid in cases of *alcaptonuria*; phenylalanine may therefore be oxidisable to tyrosine in the body. The almost complete removal of tyrosine from the mixture from casein made no difference to the growth of rats, as shown by Totani. The amount of phenylalanine in the mixture was thus probably sufficient to supply the need for aromatic nuclei. According to Abderhalden, tyrosine cannot be dispensed with.

Cystine is the only unit which contains the element sulphur, though another sulphur-containing compound may be present in proteins. The amount of cystine is not known except in a few cases, but it is estimated from the sulphur content. The need of cystine in the food has been shown most conclusively by experiments with phaseolin, the protein of the navy bean. Slow growth resulted on a diet with the protein alone, but normal growth followed the addition of 2 per cent. of its amount of cystine. Very little sulphur is present in casein. Oshorne and Mendel found that less casein was required in a diet if it were augmented with cystine: 15 per cent. casein alone gave normal growth, but 9 per cent. if cystine were added.

It is not possible to test a protein without proline, but arachin with only 1.4 per cent. was tried by Sure. This protein failed to give normal growth even after the addition of extra proline, so that its deficiency must be caused by another missing substance. It is possible that proline and glutamic acid are related units in the molecules of the proteins.

An experiment has also been made to see if the animal organism can introduce an amino group into the molecule of nor-leucine and convert it into lysine. Animals fed upon gliadin and nor-leucine did not grow just as on gliadin alone. The synthesis is thus not possible.

The whole group of simple mono-amino-acids has yet to be tested. They may not all be necessary. In cases of diabetes several can give rise to glucose; their function may be to supply energy through their conversion into sugar. Isoleucine is absent from gelatin, as recently shown by Dakin; this may be another reason for the failure of gelatin in nutrition, which is usually attributed to the absence of tyrosine, cystine and tryptophan.

#### *Comparative nutritive value of proteins.*

Few proteins show such marked deficiencies as gelatin, gliadin and zein. The different amounts of the units in complete proteins make little difference to growth if the diet contain abundance of protein. Growth is observed on the most varied proteins of animal and plant origin; but if any restriction in the amount of protein in the food be made, then the growth is lessened or inhibited. Each complete protein will thus have a definite minimum for growth. Two per cent. lactalbumin gave maintenance, 4.5 per cent. growth; 2 per cent. edestin scarcely gave maintenance, 4.5 per cent. slight growth; with other proteins 2 per cent. led to loss of weight. With a protein content of 4.5 per cent. the best growth was with lactalbumin, followed by edestin; there was no growth with casein unless supplemented with cystine, or with glycinin or squash-seed globulin. An experiment showed that a food with 9 per cent. of lactalbumin was equal to one with 12 per cent. of casein or with 15 per cent. of edestin.

#### *The nutritive value of the proteins of legumes, nuts, etc.*

The legumes contain large amounts of protein, and the chemical analysis of the proteins shows no abnormality. The proteins of the pea have been found inadequate by McCollum; it is probably due to lack of cystine, for, as stated above, phaseolin is supplemented by cystine.

The soya-bean protein is of good quality for normal growth, so also are the proteins of the peanut. The latter are peculiar in their high lysine content. The proteins of these foods require cooking so as to make them capable of being digested and assimilated.

The edestin of hemp-seed and cotton-seed is not a perfect protein with its high arginine and glutamic-acid content, but the foodstuff is largely used in America as cattle food. Its poisonous constituent can be removed by steaming or by the hot method of oil extraction.

Nut proteins have a high value on account of the high proportion of hexone bases which they contain. Normal growth has been observed on coconut press-cake, walnut, filbert, pine nut, and other nuts. Experimental work has thus confirmed the assertion of fruitarians of the high value of nuts in nutrition.

#### *The nutritive value of the mixture of proteins in cereals.*

Though the gliadin group of proteins of cereals is not adequate as source of protein in the food, it does not follow that the mixture of proteins in the grain is likewise insufficient. Wheat and maize glutenins as sole protein have been found satisfactory for growth, and may compensate for the inefficiency of the gliadins. The whole grain contains also small quantities of an albumin, globulins, and proteose. Particular attention has been paid to the nutritive value of cereals. The results are not altogether consistent; the discrepancies seem to be due to the different basal diets used by the various investigators. The results of McCollum and associates are very contradictory; they believe the inadequacy depends on improper mineral supply. Insufficiency of vitamin supply is a contributory cause. Osborne and Mendel tried not only the whole

grain, but also different commercial articles produced by milling, such as wheat flour, bran, embryo. Normal growth followed the use of whole grain, and very little difference was noticed among the various grains.

In all cases animals kept for long periods produced small or no litters of young; their health had thus been impaired. This loss of reproductive power has been observed by all workers if cereals supply the sole protein.

Wheat embryo contains the albumin, and is fully adequate; bran proteins are even of superior value. Wheat flour, pearl barley, and maize meal are not adequate.

The milling process thus removes "good" protein. Wheat flour, etc., does not contain enough total protein, but in the experiments the total was made up by adding gluten. The endosperm of the grain will only furnish protein for maintenance.

#### *Supplements to the proteins of cereals.*

Neither men nor animals consume the whole cereal, and as the endosperm does not supply enough protein in quantity or quality, it must be supplemented. The protein ratio of 1:10 needs to be increased up to 1:5. Animal proteins form the best supplements. Lactalbumin was shown by Osborne and Mendel to be the best supplement, but for some peculiar and unaccountable reason other workers do not find this protein so good. Meat, milk, eggs are almost equally efficient, but casein is of less value. In the case of maize meal and milk at least an equal part of skim milk is needed; this gives 30 per cent. of the mixture of proteins as derived from milk. Yeast protein and peanut flour are good supplements. Cottonseed and pea are inferior, whilst products like distillers' grain or vegetable albumin, which is derived from grain, are of little value. The best supplementing proteins are thus those containing the hexone bases, tryptophan, etc. which are low in amount in the cereals.

To produce normal or rapid growth it is possible that it may be more economical to use "good" proteins, which are more expensive than "bad" proteins.

#### *Quality of protein in milk production.*

Diet plays a large part in milk production. Quantity and quality of protein are the chief factors. Since neither the animal body nor the mammary gland can synthesise amino-acids, the food must contain sufficient for the manufacture of casein and lactalbumin. Hart and Humphrey have paid some attention to this question. A high milk yield of 27 lb. requires a protein ration 1:4.5; 1:6.7 is necessary for 11 lb.; 1:8.5 is not economical. If the animal is not furnished with sufficient protein it produces milk from its own tissues. Skim milk has an efficiency of 65, against 25 of a mixture of maize and alfalfa. Gluten feed (maize embryo) has 45, flax seed 61, casein 59, milk powder 60. A comparison of clover and alfalfa on the same basal diet showed a superiority of alfalfa for high milk production for 16 weeks. With another basal diet the reverse may be the case. The value will depend on the proportions of the amino-acids in the food and their correspondence to the proportions in the milk proteins. No guide is at present available from the side of chemical analysis. Little is known of the proteins of grasses and green foods. The nearest approach to the proteins of cow's milk are the proteins contained in the milk of other animals. If the milk proteins of all animals are the same in composition, the milk of one animal will be as good as that of another animal, but if not, as is most likely, the best source is the animal's own milk. Cannibalism has been proved to be the best method of feeding dogs. We approach cannibalism in the nursing of the young



on mother's milk. Milk contains ultimately the proteins of the mammary gland.

#### *Proteins and pellagra.*

The primary cause of the disease pellagra appears to be quality of protein, but at the same time insufficiency of protein together with improper salt supply may play a part.

Pellagra was not recorded in Europe before the introduction of maize into Spain by Columbus. The disease spread to France, Lombardy and eastwards, wherever maize was extensively used for food. Rousset (1866) cured it by good food, and advanced cases have been successfully treated by a generous diet (Lorentz, 1914; Willets, 1915). Goldberger cured and prevented the seasonal appearance of pellagra in lunatic asylums by increasing the meat and milk in the diet, which had previously been very deficient in this respect. Goldberger produced the disease experimentally in a squad of volunteers by a diet consisting of vegetable protein, mainly wheat, maize and rice. On a vegetable diet principally of maize, Chick and Hume produced symptoms in monkeys very like the symptoms of pellagra in man, and a cure was effected in one case by a diet containing better proteins.

Wilson, of Cairo, who investigated the outbreaks of pellagra in Armenian refugee camps at Port Said, found the diet was inadequate in energy supply and protein supply (vegetable). Thomas (1909) tested the comparative values of proteins for man, and found that meat was three times as good as maize. Wilson, calculating from Thomas' figures, determined that the refugees had a casein equivalent of 22 gm. per day. On improving to a casein equivalent of 41 gm. per day no more cases of pellagra occurred. Shortage of protein and quality of protein are thus at the root of the trouble.

### IRISH PEAT.\*

The Peat Inquiry Committee was established to inquire into and consider the experience already gained in Ireland in respect of the winning, preparation, and use of peat for fuel and for other purposes; to suggest what means shall be taken to ascertain the conditions under which in the most favourably situated localities it can be profitably won, prepared, and used, having regard to the economic conditions of Ireland; and to report to the Fuel Research Board.

Particulars of the quantity of peat produced per annum and of the peat resources of Ireland have been given previously in this Journal (*cf.* J., 1920, 213 B). From the investigations of the Committee it is apparent that if air-dried peat is to be produced at the rate of 300 to 500 tons per day, automatic machines of the Wielandt, Baumann, Persson, Jacobson, or Moore type will have to be used. By using such machines instead of hand-winning, the number of men required will be reduced to one-fourth, although the number of women and children will be slightly increased. It is possible also that a longer season could be worked with peat-machines than without, as the men working the machines can be protected from the weather. Night-work might also be undertaken, as cheap electric light would be available. Machine-cut peat dries more regularly than slane-cut peat, is less affected by rain and recovers more rapidly from its effect. This results in quicker drying and

offers the possibility of drying more peat per unit area of drying ground than when hand methods are used; and this would lead to greater economy in collection. Machine-cut peat shrinks more than slane-cut peat, with a resulting increase in density, and hence less volume is required for equal calorific value. The volume for equal calorific value may be reduced from  $5\frac{1}{2}$  to 4 or even to  $3\frac{1}{2}$  times that of coal. Machine-cut peat, owing to the breaking down of the original structure, has a more uniform distribution of the fibres, is less brittle and is better able to stand handling in transport. Peat-machines can cut for the whole depth of a deep bog, so that a more uniform material is obtained, which is very important for gas-producing or steam-raising. It is estimated that one acre of bog will yield 2600 tons of air-dried peat (25 per cent. water), and hence an output of 100,000 tons would involve the cutting away of about 38 acres per annum. To make provision for about 100 years, such a scheme would require some 3800 acres of bog or 5000 acres of land, allowing 1000 acres for the necessary drying-ground and 200 acres for room for working the peat-machines, transport and stacking. To win mechanically 100,000 tons of air-dried peat and deliver it at a distance not exceeding, say, 3 miles from the bog, would require approximately 130 men and 400 women and children for a season extending from February to the end of September. For the remainder of the year only about 35 men would be required for transport.

The Committee recommended that the State purchase a large bog of at least 10,000 acres, and provide four electrically-operated peat machines of the automatic dredger, macerator and transporter type, together with appliances for collecting, stacking and loading, and narrow-gauge tracks with electric locomotives and wagons. It also advised the erection of an electric power-station of at least 500 kw., using peat fuel and supplying power for the machines and transport. It was recommended that the bog be prepared by main drainage, by local drainage for peat-winning and local drainage for agriculture, and also that the necessary housing be provided for the employees, and office and laboratory accommodation be found. The Fuel Research Board intimated its willingness to direct the work of an experimental peat-station in Ireland and, whilst not committing itself to any definite policy, suggested as a broad programme the purchase by the Government of a suitable bog area, the establishment on that area of an agricultural colony under the control and management of the Irish Department of Agriculture, the establishment of an experimental station on the bog area under the control of a deputy director to be appointed by the Fuel Research Board, and the appointment of an Irish Advisory Committee in connexion with the station. The Board's contribution to such an establishment would be limited to buildings and machinery (£31,000), plus £6000 for contingencies, plus running expenses for 4 years (say £14,000), making a total of £54,000.

An agricultural sub-committee appointed by the Department of Agriculture and Technical Instruction for Ireland reported that an agricultural colony is not, and cannot be, complementary to a scheme for the mechanical winning of peat fuel, and that the reclamation of virgin bog would not be remunerative under the most favourable conditions, although the reclamation of cut-away bog might be remunerative provided (a) that the cut-away bog afforded facilities for drainage, (b) that a sufficient depth of peat were left by those in charge of the peat-fuel winning operations, and (c) that either the stratum underlying the peat residue consisted of suitable mineral matter, such as marl, limestone, gravel or clay with an admixture of sand, or that one or more of these materials were present in close proximity to the cut-away bog.

\* The Winning, Preparation, and Use of Peat in Ireland: Report and other Documents. Fuel Research Board, Department of Scientific and Industrial Research. H.M. Stationery Office, 1921. Price 1s. net.

The sub-committee was also very sceptical as to the cost of the bog, the cost of housing the labour, cost of live-stock, implements, and incidental expenditure on roads, fences, etc.

Prof. Pierce Purcell has been appointed Peat Investigation Officer to the Fuel Research Board, and further inquiries and research are proceeding.

## SOCIETY OF CHEMICAL INDUSTRY.

### ANNUAL MEETING, 1921.

#### SULPHURIC ACID IN CANADA.

Those who follow the development of Canadian chemical industry, and in particular those who are attending the Annual Meeting in Montreal, will be interested in the following short history of the manufacture of sulphuric acid in the Dominion.

Following the discovery of petroleum in the Ontario peninsula in 1863, plants were erected at Hamilton, Woodstock, London, and Petrolia, Ontario, and realising the economy of producing the acid in close proximity to the oil refineries the Canadian Chemical Manufacturing Co. built the first sulphuric-acid works at London. The plant was initially quite a small one, compared with such plants to-day, and consisted of one sulphur burner of the Blair type, two lead chambers, and a concentrating system of lead pans with about twenty glass bottles which were termed retorts. The point of desired concentration was determined by the appearance of white fumes over the surface of the acid, or by observing the clarification produced after adding a pinch of sawdust. Under this treatment the acid first became black, but on attaining a specific gravity of 1.825 it became transparent. This was indeed a crude and inexact method, but it accorded with the practice of that time. Sicilian sulphur was the raw material employed and the amount of acid produced was from 50 to 60 tons per month. When manufacturing first began the water necessary for the reaction was placed in the bottom of the chambers, and this method was followed for several months before the idea of supplying it as injected steam was introduced. As might be expected this was followed by a much larger yield of acid. Not long after, pyrites was used instead of sulphur. The plant was destroyed by lightning a few years later, and when rebuilt on a larger scale the burners were converted to the sulphur type. In 1887 the factory was burned down, and rebuilt with three lead chambers, each 120×20×18 ft., which were not housed in buildings as their predecessors were. The retort system of concentration, in which breaks were frequent, was superseded and improved by the use of the Fanre and Kessler type, and production was increased to about 10 tons per day. The factory was dismantled and removed in 1904.

The second Canadian plant was built in 1870 by an English syndicate which purchased land at Petrolia and London, Ont., sank oil wells, and erected at London an oil-refinery and sulphuric-acid works known as the Ontario Chemical Works. The plant consisted of two small chambers with glass-retort concentration and produced from 35 to 40 tons per month; it was absorbed by the company mentioned above, and in 1888 was dismantled.

In the early 'seventies, when pyrites was discovered within about four miles of Brockville, Ont., the Brockville Chemical Co. erected a plant of four chambers, but before actual manufacturing began the buildings were blown down by an unprecedented wind.

After being rebuilt the plant worked successfully for a few years, but its career came to an abrupt end owing to the pyrites deposits becoming exhausted.

The Nichols Chemical Co. built a large plant at Capleton, Quebec, about the year 1887, and in 1895 the Victoria Chemical Co. established a plant at Victoria, British Columbia, which is still in existence, and produces acid of 60° B. In 1901 the Dominion Iron and Steel Co., then known as the Dominion Coal Co., started a small plant at Sydney, Nova Scotia, which was replaced by a larger one in 1911. In 1906 the chamber system of the Nichols Chemical Co. at Capleton, Que., was replaced by the contact system. Other contact plants were established by the same company at Sulphide, Ont., in 1907 and at Barnet, British Columbia, in 1909. These are the only contact plants in operation in Canada at the present time. In 1911 the Grassli Chemical Co. erected a chamber plant at Hamilton, Ont., which concentrates the acid to 66° B. by the cascade system. In 1912 the Algoma Steel Corporation, of Sault St. Marie, Ont., erected a Faulding high-chamber plant to manufacture sufficient acid to sulphate the ammonia in its coke-oven gas. This plant is also still operating. Other acid plants of more recent date include that of the Consolidated Mining and Smelting Co., at Trail, British Columbia, built in 1916, and during the war the Aetna Chemical Co. erected a chamber plant at Drummondville, Que., which may be dismantled. The Chemical Products, Ltd., has a plant at Trenton, Ont., which was taken over from the British Chemical Co. after the war and now specialises in the production of acid phosphate.

### NEWS FROM THE SECTIONS.

#### SHAWINIGAN FALLS.

On May 25 Dr. G. S. Whithy, assistant professor of chemistry, McGill University, gave an address on Rubber to the members of this Section.

At the outset the lecturer drew attention to the rapid growth of the rubber-plantation industry during the decade 1909-19, the total production of raw rubber having increased about sixfold, and that of plantation rubber from approximately 3000 to 300,000 tons per annum. Methods of preparing plantation rubber were outlined. Special attention was drawn to the important influence on vulcanisation of the small amount—about 5 per cent. in all—of non-caoutchouc substances present in the raw rubber, the influence of the resin being particularly marked when the rubber is compounded with litharge. Reference was made to the investigations now being conducted at McGill University on the chemical nature of the rubber "resins."

With the aid of lantern slides, the processes employed in the making-up and vulcanisation of rubber articles were outlined. The influence of the chief compounding ingredients was considered, the growing importance of organic accelerators of vulcanisation being emphasised. Data were given showing that such accelerators may not only increase the speed of vulcanisation, but that they may also confer improved physical properties on the vulcanisates; and further, that different accelerators may exercise favourable effects in different directions, e.g., one may improve tenacity whilst another improves extensibility. Mention was made of the work of Wiegand on the influence of inorganic compounding ingredients. The uniqueness of the mechanical properties of vulcanised rubber was brought out by consideration of the stress-strain curve, of the magnitude and character of hysteresis and elastic after-effects in

rubber, and of the relation between thermal and tensile effects. Experiments illustrating these effects were shown.

#### LONDON.

At the meeting held on June 6, Mr. J. L. Baker presiding, a paper entitled "Industrial Respirators," by Dr. L. Levy and D. W. West, was read by the former.

A respirator consists essentially of two parts: a face-piece and an absorption apparatus. The face-piece may consist only of a mouthpiece and nose-clip, but this form causes much discomfort and affords no protection to the eyes. The second and more recent form is a complete mask of rubber fabric with eyepieces of "Triplex" glass so arranged that the incoming air passes over the inner surface of the glass, thus preventing dimming by moisture from the breath. This form requires neither the use of a mouthpiece nor of a nose-clip, and may be worn for a much longer period than the former type. The purifying canister is of the form used in the small military box-form respirators, and the substances employed for absorption should have a high capacity and rate of reactivity for the gases to be removed from the inspired air. The absorbents used vary according to the toxic substance to be dealt with; crystallised copper sulphate, highly activated charcoal, and a mixture of alkali and charcoal powder are used, respectively, for ammonia, hydrocarbon and other neutral gases, and acid gases. A satisfactory means of removing smoke and solid particles is a mask of fine felt fitted with an efficient non-return valve for the egress of the expired air. No absorbent for carbon monoxide is known which is suitable for use in a respirator, and, therefore, the catalytic oxidation of this gas by oxides is employed. But this method has certain disadvantages, e.g., the harmful effect of moisture upon the catalyst (which may be overcome by a preliminary passage of the gases through calcium chloride), the absorption of a portion of the atmospheric oxygen to oxidise the carbon monoxide, and the absence of any indication of failure in the respirator. The last-named defect may be remedied by using an upper layer of iodine pentoxide and sulphuric acid which, when any carbon monoxide unoxidised by the catalyst is absorbed, liberates free iodine vapour that acts upon the throat of the wearer and thus indicates the breakdown of the purifying medium. The tests used in determining the efficiency of different forms of respirator were described.

Professor K. G. Naik then read a paper on "The Gold and Silver Thread Industry of India," which prior to the war had become nearly extinct owing to the dumping of German-made material upon the Indian markets. During the war this source of supply was cut off and the native industry resuscitated. Most of the work is done by hand—the little machinery that is employed is of a primitive kind. Silver bars, 9 in. long by 3 in. square, are coated with 0.5–1.0 per cent. of gold by hammering, and the bars are further hammered out to a length of thirty inches. The resulting rods are then pulled through a series of draw-plates until each ounce is converted into a fine wire some 2000 to 4000 ft. long. After polishing on a pad of leather or cloth, the wire is flattened into ribbon by passing between rollers, and is then wound into a helix upon a rotating needle, or wound spirally around cotton or silk threads. Spangles are produced by cutting the helix lengthwise and beating the rings so obtained with a special kind of hammer. A method devised by the author for plating the fine silver wire by electro-deposition in a gold-cyanide bath was described, and lantern slides of various stages in the manufacture as well as numerous samples of the different products were exhibited.

## MEETINGS OF OTHER SOCIETIES.

### THE PHYSICAL SOCIETY.

At the meeting held on June 10, Sir Ernest Rutherford lectured on "The Stability of Atoms."

The study of the scattering due to  $\alpha$  particles has led to the view that the atom consists of a minute positively-charged nucleus surrounded by a number of rings of negatively-charged electrons. A direct verification of Moseley's law (the nuclear charge is represented by the atomic or ordinal number of the atom), to an accuracy of about 1 per cent., has recently been given by Chadwick for the elements copper, silver, and platinum. The atomic radius of the atom of a heavy element such as uranium (ordinal number 92) is of the order  $10^{-8}$  cm., and the corresponding nuclear radius is  $7 \times 10^{-12}$  cm.; in the case of lighter elements, such as helium, the nuclear radius is about  $3 \times 10^{-11}$  cm. The only promising line of attack whereby a permanent effect may be produced upon the atomic structure of an element appears to be that afforded by the impact of swiftly moving  $\alpha$  particles with the atomic nucleus; no permanent effect upon the atomic structure is to be anticipated from effects produced in the rings of negatively-charged electrons. The great stability of the atomic structure arises from the minute dimensions of the nucleus, and the manner in which the nucleus is guarded by the rings of electrons. It is, however, possible by the bombardment of swiftly moving  $\alpha$  particles to effect an occasional collision of the particle with the atomic nucleus of an element, more especially a gas, and in this manner, the lecturer has shown that in the bombardment of nitrogen, hydrogen is apparently ejected from the nucleus. The nature of the ejected particle is ascertained from its range of ejection. Recently the method has been extended to the heavier elements such as aluminium, and the surprising result has been obtained that in such cases and also in the case of nitrogen, particles are ejected having a range greater than 80 cm., the range of ejection of the hydrogen particle being 28 cm. The possibility of the ejected particles being due to contamination with hydrogen is definitely ruled out by the method of experiment. Thus the possibility of attacking the nucleus has been advanced a stage, and the lecturer concluded by remarking that the recent work of Shimizu has shown that the nucleus would be very readily disintegrated, with the liberation of prodigious atomic energy, provided the  $\alpha$  particle were able to approach sufficiently near the atomic nucleus.

### THE ROYAL INSTITUTION.

Sir J. J. Thomson delivered the Friday Evening Discourse on June 17 on "Chemical Combination and the Structure of the Molecule." After a brief review of the electron theory, the lecturer outlined his theory of atomic structure. In this it is assumed that the force between the central positively charged nucleus of the atom and the electrons is not in accordance with the inverse square law, but assumes a series of periodic attractive and repulsive values as the distance apart increases. Eight is then the greatest number of electrons that can be in stable equilibrium without reproducing electronic distributions characteristic of atoms containing fewer electrons, a result in accordance with the Periodic Law. It was pointed out that the periodicity of atomic sizes as deduced from this theory was in accord with the results of the experiments of Bragg but differed somewhat from the periodicity as exhibited in Lotbar Meyer's table. The theory indicates the possibility of the formation

of compounds of the inert gases. Chemical combination between two atoms is supposed to occur as the result of the coalescence of the cellular structures of electrons and positive nucleus constituting the atoms. This coalescence may be effected in more than one way. A ready explanation is thus afforded of the existence of, *e.g.*, a combination of carbon and oxygen both as carbon monoxide and as the radicle=CO. The arrangement of eight electrons at the corners of a cube with a positively charged nucleus at the centre constitutes an essentially stable system. Such cubes can be combined geometrically in a variety of ways of which two were instanced by the lecturer. In the first, the cubes are combined so as to have a face in common, and in the second a side is common to them. Analysis shows that the second is the more stable arrangement, and in this manner is explained the existence of the very extensive series of carbon chain compounds of the generic formula  $(CH)_n$ , compounds of this type being known up to the value  $n=60$ . The series formed by cubical structures having a face in common is a very restricted one and is known only up to  $C_6H_6$ . The benzene ring contains thirty electrons, each carbon atom containing four and each hydrogen atom one. To constitute such a ring from six cubical structures of eight electrons requires eighteen electrons to be held in common. Clearly this can be effected by constituting the structure of six cubes having alternately a face and a side respectively in common. Such a mode of constitution affords an explanation of the existence of alternate single and double bonds between successive carbon atoms in the benzene-ring structure. In this connexion it was pointed out that theory led to the conclusion that the equilibrium figure of eight electrons about a central positive nucleus was not a cube but what might be termed a "twisted cube" formed by twisting one face through half a right angle relative to the opposite and parallel face about an axis perpendicular to the faces. A symmetrical benzene-ring structure would result from the coalescence of triangular faces of six such twisted cubes, whereby the economy of eighteen electrons in the structure would be effected in a uniform manner.

At the conclusion of the lecture, the Duke of Northumberland, president of the Institution, presented on behalf of the members a gold loving cup to Sir James and Lady Dewar on the occasion of their golden wedding.

#### THE ROYAL SOCIETY.

At the meeting held on June 16, a paper entitled "The Ratio of the Specific Heats of Air and of Carbon Dioxide," by Prof. J. R. Partington, was communicated by Dr. J. A. Harker.

The ratio of the specific heats,  $\gamma = c_p/c_v$ , was determined by the method of adiabatic expansion. The gas was contained in a 120-litre vessel, and the temperature-change immediately after expansion followed by a platinum thermometer, with compensating leads of wire 0.001 mm. diameter, the resistance of which was observed by an Einthoven string galvanometer of 0.01 seconds period. The fundamental temperature measurements were made with a mercury thermometer. The results were calculated by the characteristic equation of D. Berthelot, so that deviations from the ideal gaseous state were allowed for. The final results, which are believed to be accurate to 1 part in 1000, are:  $\gamma$  for air at  $17^\circ C. = 1.4034$ ;  $\gamma$  for carbon dioxide at  $17^\circ C. = 1.3022$ . From these numbers the specific heats of the gases were calculated by Berthelot's equation, with the following results:  $c_p$  for air at  $17^\circ C. = 0.2387$  cal.;  $c_p$  for carbon dioxide at  $17^\circ C. = 0.1996$  cal. All the values refer to atmospheric pressure.

#### SOCIETY OF GLASS TECHNOLOGY.

The last meeting of the session 1920-21 was held in the University of Sheffield on June 15, Dr. Morris W. Travers presiding. Dr. P. G. H. Boswell, professor of geology in the University of Liverpool, was elected the first honorary member of the Society.

A discussion was then held on "Oil-firing in the Glass Industry." The president showed by means of thermochemical calculations that the fuel efficiency of a furnace in which the oil was burned with air at the ordinary temperature, and that of a furnace in which coal was first gasified by air with only little steam, the gas being burned with air at the ordinary temperature, were practically identical. That non-regenerative glass tanks fired with oil were preferred to those fired by gas was probably due to the fact that small gas-fired tanks were inefficient. In oil-firing practically the whole of the air could be preheated, but in gas-firing only the secondary air could be preheated, so that a regenerative oil-fired furnace was more efficient than a furnace in which the secondary air was preheated. Other conditions being equal, the ratio of the efficiency of the oil and gasified-coal systems was as 1.37 to 1. Neglecting other factors, the results might be summed up as follows:—

	Net calorific value.	Cost in London per ton.	Efficiency of combustion.	Relative cost.
Oil ..	10,500	105s.	1.37	1.50
Coal ..	7,420	45s.	1	1

Mr. J. Connolly remarked that he had run an oil glass-furnace for four years, which had worked with fewer breakdowns and stoppages than any other method. At the end of that period, however, the price of oil was raised and its use was abandoned. The adaptability of oil to glass work had been demonstrated and was past argument, but he did not think they would get a higher efficiency with oil than with coal so long as there was a great difference in price.

Mr. J. H. Steele thought that oil had not been given a fair chance in the glass industry; his experience of it was very satisfactory. It was unusual, however, to find a glass furnace properly adapted to oil-consumption. He had known better results to be obtained from the use of crude oil or creosote than from fine petroleum of high calorific value, a fact which indicated that the furnaces in use were not satisfactory.

Mr. F. G. Clark spoke of the use of "glory holes" fired by oil. The best results were obtained from crude creosote oil, partially heated, and compressed air at about 15 to 20 lb. per sq. in. In making 8-ounce bottles on a  $7\frac{1}{2}$  hour shift some 3.92 gallons of oil were used, at a cost of 4s. 3d. per shift. When two chairs were working at one "glory hole," the cost was about 2s. 2d. per shift. Gas did not yield the temperature desired, and creosote oil gave a fuller and "fatter" flame than petroleum. Consequently the neck of the bottle was heated up more uniformly and the product had a better "finish."

Mr. G. Lawton described a simple oil-burner that had yielded good results. In one power-station one ton of oil was apparently equivalent to about 2½ tons of coal.

Mr. J. Venn Stevens pointed out the advantages of oil-fuel for glass furnaces. It was clean in use, the flame came into direct contact with the glass without producing any discoloration or deterioration, and a uniform temperature could be maintained.

Mr. A. Bayliss remarked that the "Aquitania" had in the stoke-hole 350 men when fired with coal, but only 84 with oil-fuel. With solid fuel the efficiency might range from 50–65 per cent., but with oil-fuel it might be as high as 85 per cent. In addition, ignition and extinction were instantaneous. The freedom from sulphur in the gases

formed by combustion was an important point for glass workers.

Mr. W. H. Casmev referred to the very great potentialities of oil when mixed with inferior qualities of solid fuel. In one case, by taking 3 tons of coke breeze (at 12s. 6d. per ton, calorific value 10,000 B.Th.U.) and one ton of oil-fuel, a fuel of 12,000 B.Th.U. was obtained costing 37s. 4d. per ton, capable of giving an evaporation of 530 lb. per ls., or nearly 2000 lb. more evaporation per ton than with coal at 40s. per ton.

Mr. W. J. Rees thought it was very necessary to obtain reliable data showing how efficient coal-firing compared with efficient oil-firing.

Prof. W. E. S. Turner exhibited and explained a diagram showing the proposed design of a combustion-chamber for the oil-firing of a glass tank furnace. He also read a communication from a firm in which it was stated that there were very few oil-fired pot furnaces in this country. The few that had been tried had not been entirely successful, the chief trouble being the cracking of pots. This was due to excess of heat in one particular place on the pot, causing unequal expansion. A method of avoiding this was suggested.

#### BRITISH PHARMACEUTICAL CONFERENCE.

The fifty-eighth annual meeting of the British Pharmaceutical Conference was held at Scarborough from June 13 to June 16 inclusive, under the presidency of Major E. Saville Peck, of Cambridge.

In his presidential address Major Peck demonstrated the need for higher education. Pharmacy, if it is to progress, must move forward with the advance of general education and of applied science, and if it is to take up its position with other professions it must bring its final qualifications up to University and Continental standards. He advocated the institution of a fellowship examination which would entitle the holder to call himself a clinical analyst or clinical pharmacist, or registered clinical assistant of the Pharmaceutical Society, and urged the inclusion of practical physiological chemistry and bacteriology (including chemical microscopy) among the compulsory subjects. In British pharmacy to-day there is need of a closer union of purpose, counsel, and activity of all those who are working for a full realisation of the profession.

Among the papers communicated to the Science Section was one on "The Determination of Formaldehyde and Paraformaldehyde in Tablets," in which Messrs. Norman Evers and C. M. Caines described a method based on a comparison of the colour produced by the addition of Schiff's reagent to a solution of a tablet containing formaldehyde, with that produced by the same reagent on a standard solution of formaldehyde of known concentration. The accuracy of the estimation by the authors' method is not influenced by the presence of menthol, milk sugar, or cane sugar usually present in the tablets.

In a paper on "The Titration of Certain Alkaloids" Mr. Norman Evers showed that bromophenol blue is the most suitable indicator to use in the titration of morphine, atropine, and the neutral salts of quinine, but for an acid salt of quinine, methyl red is preferable. A method for the titration of ammoniated quinine for the combined determination of quinine and ammonia was described.

Mr. A. J. Jones read a paper on "The Quantitative Determination of Minute Quantities of Bromide in Saline Residues, and in a Mixture of the Halogens." The author advocates the use of a method which is a development of Denigès' process. The method is applicable when dealing with quanti-

ties of about 0.1 mg. of bromide existing alone or in admixture with iodide and chloride in water residues, or the residue from the ignition of any organic substance.

In a paper on "The Determination of Nitrates in Bismuth Salts by means of Titanous Chloride and Devarda's Alloy," Mr. T. McLachlan showed that the British Pharmacopœia method for the estimation of nitrates in bismuth carbonate is unreliable. The method for estimating the nitrate by reducing with titanous chloride, distilling with steam the ammonia formed, collecting in acid, and titrating back, was found tedious, but satisfactory results were obtained by using Devarda's alloy in alkaline solution in the presence of a small amount of alcohol. The author's method can be used for estimating the amount of nitrate in any bismuth salt excepting those in which ammonia is present.

In a paper on "The Determination of Small Quantities of Phosphate in Glycerophosphates," Mr. J. L. Lizius referred to the need on the biochemical side for a simple, rapid and accurate method for the estimation of small quantities of phosphoric acid, and on the analytical side to the need of a method for estimating small amounts of phosphate, present as impurities in glycerophosphates. The author's method is a tintometric one based upon a comparison of the colour obtained by adding nitric acid and ammonium molybdate to a standard solution of phosphoric acid and to a solution of the glycerophosphate under examination.

Mr. E. C. Davies communicated details of a process devised for "The Assay of Colchicum by means of Phosphotungstic Acid." Phosphotungstic acid precipitates colchicine in a bulky form in a 1.5 per cent. solution of sulphuric acid. The author's method may be summarised as follows:—The drug having been exhausted with alcohol, and the alcohol recovered, the colchicine is taken up with water, shaken out with chloroform, again taken up with hot water, precipitated as phosphotungstate, from which the colchicine is liberated either by chloroform and alcohol, or by chloroform, alkali and alcohol. The special advantages of the method described is that the alkaloid is obtained in a greater degree of purity than by other processes.

Messrs. C. E. Corfield and G. R. Boyes, in their paper "An Examination of Commercial Bismuth Phenate," showed that some samples of the salt contained little or no phenol. They suggested a method of preparation yielding a product containing 14.76 per cent. of phenol, this being the maximum content which it was found possible to obtain.

The following papers were also communicated to the meeting:—"The Ether-solubility of Scammony Resin"; "The Analysis of Theobromine Sodium Salicylate"; "A Note on Liquefied Carbolic Acid"; "The Quantitative Determination of Sodium Arsenate"; "The Assay of Atropine and Cocaine in the B.P. Ointments"; "The Approximate Estimation of Commercial Cresol in Lysol"; "A Contribution to the Study of Digitalis"; "A Note on the Official Method for the Determination of Hydrocyanic Acid"; "The Nature and Composition of Irish Moss Mucilage"; "The Action of Heat and Moisture on the Activity of Ergot and Liquid Extract of Ergot"; "An Investigation of some of the Factors which may determine the Keeping Qualities of Infundibular Extract"; "The Volumetric Determination of Arsenic Acid and Arsenates."

At the closing session of the Conference, Prof. H. G. Greenish, professor of pharmacæutics at the Pharmaceutical Society's School of Pharmacy, was elected president of the Conference for the ensuing year, and an invitation was accepted from the Nottingham Pharmaceutical Association to hold the annual meeting of the Conference at Nottingham in 1922.

## PERSONALIA.

The University of Wales is to confer the honorary degree of Doctor of Science upon Sir J. J. Dobbie.

Dr. Eric K. Rideal has been appointed to the Humphrey Owen Jones lectureship in physical chemistry at Cambridge University.

Prof. S. P. L. Sørensen of the Carlsberg Laboratory, Copenhagen, has been awarded the Leblanc Medal of the Société Chimique de France.

The degree of Doctor of Science of the University of Manchester is to be conferred upon Messrs. F. W. Attack, Colin Campbell, and R. G. Fargher.

Dr. W. J. Jones, senior lecturer in chemistry in the University of Manchester, has been appointed to the chair of chemistry in University College, Cardiff.

Prof. J. von Braun, of Berlin, who was recently appointed director of the Chemical Institute in Frankfurt, has been called to the chair of chemistry in the University of Vienna.

Dr. C. F. Chandler, who was president of this Society in 1899—1900, and Dr. William H. Nichols, president in 1904—1905, have been elected honorary members of the American Chemical Society.

Dr. E. W. Washburn, head of the ceramic department of the University of Illinois, has been appointed editor of the *Journal of the American Ceramic Society* in succession to Mr. H. F. Staley.

The Nichols Medal, which is presented annually for the best original paper published in any of the journals of the American Chemical Society, has been awarded for 1920 to Dr. Gilbert N. Lewis for the paper on "The Third Law of Thermodynamics and the Entropy of Solutions and of Liquids," by himself and Mr. G. E. Gibson.

Dr. Robert Robinson has been appointed to succeed Prof. J. C. Irvine as professor of chemistry and director of the chemical research laboratory in the University of St. Andrews. Dr. Robinson was for several years professor of organic chemistry at Liverpool University and in 1920 was appointed director of research to the British Dyestuffs Corporation, Huddersfield.

We regret to record the death on June 21, at the age of eighty, of Mr. David Brown, late senior partner of J. F. Macfarlan and Co., manufacturing chemists, Edinburgh, and an original member of this Society.

The late Mr. William J. Chrystal, chemical manufacturer, of Shawfield Works, Rutherglen, and an original member of this Society, bequeathed £10,000 each to the University and the Royal Technical College, Glasgow.

The death is announced of Dr. F. P. Dewey, head chemist of the Mint Bureau, Treasury Dept., Washington, D.C., who joined this Society in 1889; also of Ambrose Monel, at the age of 47, the inventor of "Monel" metal. Monel was a graduate of Columbia University and became the first president of the International Nickel Co. in 1902, at the age of twenty-nine.

Dr. Ludwig Knorr, professor of chemistry in the University of Jena, who died on June 4 in his 62nd year, is perhaps best known for his discovery of antipyrin and for his researches in the pyrazole group, but his work on the constitution of morphine, on new methods of synthesising amino-alcohols, quinoline and pyrrole, and his investigations on keto-enol equilibrium will long be remembered as outstanding achievements in the sphere of organic chemistry.

## NEWS AND NOTES.

## UNITED STATES.

**The Bureau of Mines Cryogenic Laboratory.**—The new Cryogenic Laboratory of the U.S. Bureau of Mines was opened by Madame Curie on the occasion of her recent visit to Washington. The main work of the laboratory will be to obtain fundamental scientific data, especially such as will enable the Government plants to produce helium more efficiently and at lower cost. Its second and subsidiary object is to provide facilities for a limited number of American scientists who wish to carry out special low-temperature investigations, particularly along lines not covered by the work of the technical staff of the Bureau of Mines. The laboratory is directed by Dr. R. B. Moore, chief chemist to the Bureau of Mines.—(*Chem. and Met. Eng.*, May 25, 1921.)

**Sugar Consumption in 1920.**—According to statistics published in the *American Sugar Bulletin*, the consumption of refined sugar in the United States in 1920 was 4,075,173 tons, as against 4,126,673 in 1919, the per capita consumption in the two years being 86.35 and 87.45 lb., respectively. The total sugar consumed, the United States produced 144,000 t. cane, 900,000 t. beet, and 20,000 t. of other sugars. A feature of the imports is the large supply obtained from Java and other countries that pay full tariff rates, rendered necessary by the heavy consumption of home-produced sugar in the autumn of 1919 and the decrease in the Cuban crop of 1919-20. There was a decrease of 268,106 t. in the exports (roughly 400,000 t.) so that a larger amount became available for home consumption. Stocks in hand on January 1, 1921, were 1,093,545 t., the stocks of domestic cane beet, and imported sugar being 715,862 t. greater than in 1919. This stock is about equal to the crop of Cuban sugar still held in Cuba, and therefore by no means excessive (*cf. J.*, 1921, 173).—(*Ind., Tr. J.*, May 20, 1921.)

**Cadmium in 1920.**—Further progress is reported in the output of cadmium in the United States. The production in 1920, 129,283 lb. of metal, valued at \$151,261, being nearly 30 per cent. greater than in 1919. The production of cadmium sulphate amounted to 32,133 lb., valued at \$37,133, against 31,197 lb. in 1919. Since 1910 the American production has steadily increased, and Germany with a production of 85,395 lb. in 1919, now takes second place. The metal has not been imported into the United States since 1915, but about 40,000 lb. was exported in 1918, chiefly to France (*cf. J.*, 1920, 437 *x*). A comparatively new use for cadmium is in making small-arms ammunition. A cadmium band on the hard-jacketed cartridge ball takes the rifling with little wear on the barrel and thus prolongs the life of the gun. Electroplating with cadmium offers promise of expansion and as the metal is a better rust-preventive than nickel, it might replace nickel for this purpose where there is less difference in price. It is suggested that as cadmium tarnishes more readily than nickel the latter metal might be deposited as a first coat of cadmium. A commercial process for electroplating iron and steel articles has been developed by the Udylite Process Co., Kokomo, Ind. The pigment industry offers a good protective field for the consumption of cadmium compounds, chiefly cadmium yellows and "cadmium lithopone."—(*U.S. Geol. Surv.*, May 12, 1921.)

**Bromine Production in 1920.**—According to a preliminary report of the U.S. Geological Survey, the output of bromine in 1920 was 518.1 long tons valued at \$745,881, a decrease of 37 per cent. in quantity and nearly 40 per cent. in value compared with 1919. The production, however, was greater



than in any year prior to 1918 (cf. J., 1921, 173 n). The average price per lb. received by the producers rose from 13 cents. in 1910 to \$1.31 in 1916 and was 64 cents. in 1920, which is more than three times the average price in 1913.—(Official.)

#### FRANCE.

**Industrial Notes.—Chemical Industry.**—Owing to the continuance of the trade depression, more factories are closing down, the latest being those of the Société Normande de Produits Chimiques. The situation, however, is held to be merely temporary, and preparations are being made for an economic revival. Thus the Compagnie Nationale de Matières Colorantes et de Produits Chimiques is organising the production of dyes at its factory at Villero-Saint-Paul, north of Creil. The factory covers an area of 111 acres, and is divided into three groups; the first has been producing indigo for some time; the second has just started to manufacture alizarin; and the third is preparing to produce indanthrene. It is anticipated that the output of these three products and their derivatives will not only cover home requirements, but leave a surplus for exportation; and it appears that an outlet has already been found in China. More than 75 chemists are employed and, apart from the existing works laboratories, a large central research laboratory is to be established this year. The company has allocated 250,000 francs for the furtherance of research in the chief technical universities. The Claude synthetic-ammonia process is likely to be established shortly in the Landes district of south-west France, where coal, electric-power, and salt are available. It is intended to produce ammonium chloride for fertilising purposes.

**Metallurgy.**—The export of ferrous scrap is now free and the 20 per cent. *ad valorem* duty has also been abolished. Stocks of iron ores are considerable, and in a few mining areas extraction has been topped. Some producers still hold fairly large supplies of pig iron, but the stocks of others are entirely depleted. The price of hematite pig iron has been fixed at 450 fr. per metric ton as from June 1, which represents a reduction of 85 fr. on the price at April 1 last. Production of steel has been reduced and stocks, especially of heavy steel, do not find a ready market.

**Coal.**—Great activity prevails at the chief mining centres, as demand is reviving owing to depletion of stocks consequent on the continued export of coal to England. Thus the stocks of the East, State, and North railways are 50 per cent. lower than in April. The mines devastated during the war produced 392,045 t. in April or 27,750 t. more than in March.

**Power and Transport.**—The Senate has approved a scheme to utilise the Rhine for the production of 60,000 h.p., for making it navigable over a distance of 500 km. and enabling irrigation to be applied to an area of 494,000 acres. The cost is estimated at 3000 million fr., and the annual revenue to accrue to the State at 300 million fr.

The railway system in Algeria is to be extended during the present year in order to open up the various mining areas that have been discovered in recent years; about 1539 million fr. will be spent on new construction and on repairing existing lines.

**Clove Production in Madagascar.**—Practically all the cloves exported from Madagascar are produced on the island of St. Marie, off the north-east coast, only some 10 to 15 metric tons being grown on the mainland. The exports, mainly directed to France, were 143 t. in 1918 and 499 t. in 1919; the 20-21 crop amounted to only 20 t., although 87 t. is exported in December, 1920. The outlook for the 1921-22 crop is stated to be good.—(U.S. Com. p., Apr. 23, May 7, 1921.)

**The Graphite Situation in Madagascar.**—According to a statement published by the Madagascar Service of Mines, the production of graphite in Madagascar in 1920 was between 4000-5000 metric tons (1919, 4000-5000 t.; 1918, 16,000 t.; 1917, 35,000 t.). Exports during the year amounted to 14,919 t., of which 4449 t. went to England, 2127 t. to the United States, 51 t. to Belgium, and the remainder to France. Stocks at the end of the year were about 24,000-25,000 t., nine-tenths of which consisted of flake and one-tenth of amorphous carbon. Three-quarters of the flake graphite in stock averaged 80-90 per cent. carbon, the tendency now being to turn out a quality averaging 90 per cent. and above owing to the small demand. About 20 per cent. of the production is treated mechanically, some half-dozen plants being equipped with drying and separating machinery (cf. J., 1921, 110 n).—(Bd. of Trade J., May 26, 1921.)

#### SOUTH AFRICA.

**Eucalyptus Bark as a Cork Substitute.**—A correspondent writing in the *South African Journal of Industries* of March-April, 1921, draws attention to the value of the thick bark of the gum tree (*Eucalyptus sideroxylon*) as an insulating medium for refrigeration plants. A test carried out by Messrs. Sykes and Co., Ltd., of Johannesburg, showed that the insulating property of eucalyptus bark, when kept dry, was about equal to that of granulated cork, the actual figure obtained being 1.32 B.Th.U. per sq. ft. per degree difference in temperature per 24 hrs., using insulation 6 in. thick. As the bark is at present a waste product, the suggested new use should be worth consideration.

#### AUSTRALIA.

**Industrial Notes.**—The Hoskins Iron and Steel Co., Ltd., which owns large iron and steel works at Lithgow N.S.W., has purchased 380 acres of land at Port Kembla, near Wollongong, N.S.W., as a site for additional steel works. The estimated expenditure is approximately £2,000,000, and it is intended to purchase as much as possible of the plant and equipment in the United Kingdom.

Mr. A. Gibson, who is now exploring Papua (New Guinea), has applied to the Australian Government for a concession to exploit the nipa palm in the Fly River District. He guarantees a production of 500 tons of paper pulp per week and 3 million galls. of motor spirit per annum, and is reported to be willing to expend £300,000 on the erection of a plant and to employ 60 whites and 1500 natives.—(Official.)

#### JAPAN.

**The Dye and Colour Industry.**—The trade depression has affected the Japanese dye industry so adversely that at the beginning of this year only the larger and well-established factories remained in operation, and even these are now in a difficult position owing to growing German competition both abroad and at home. Nevertheless considerable progress has been made in the production of the chemicals required in dyemaking. Caustic soda and alkali of good quality are produced in fair amounts; acetic acid, acetone, nitric acid, ammonia, and glycerin are being made; nitrogen fixation and the production of soda ash have yet to be undertaken. The expectation of the *ad valorem* duty (35 per cent.) on all imported dyes was the cause of an abnormal increase in imports during 1920, so that the market became overstocked. The proposal to amalgamate the dye factories met with opposition and was not consummated. It is reported that the Japanese Government intends to stimulate dye-production by assisting the Japan Dye Manufacturing Co., which was floated at the beginning of the war. The progress made by the Japanese dye industry is

shown by the following statistics of imports in 1913 and 1919:—

	1913. lb.	1919. lb.
Indigo, natural . . . . .	27,650	1,030,812
Indigo, synthetic . . . . .	2,203,969	521,316
Logwood extract . . . . .	963,624	1,846,605
Alizarin . . . . .	212,661	—
Aniline . . . . .	9,816,613	4,364,830
Coal-tar dyes, other . . . . .	—	114,935
Ultramarine blue . . . . .	324,930	183,960
Prussian blue . . . . .	87,662	—
Cobalt oxide . . . . .	40,439	114,414
Liquid gold, silver and platinum . . . . .	59,997	2,133

Of a total import of 13,737,545 lb. of dyes in 1913, over 80 per cent. came from Germany, but in 1919, out of a total of 5,943,371 lb., the United States supplied 83 per cent.; in 1920, the origin of the 4,479,863 lb. of coal-tar dyes imported was as follows:—United States 67, Germany 17, United Kingdom 6, and France 4 per cent. During 1920 Japan exported 5,595,140 lb. of coal-tar dyes, but it is not known whether they were made in the country or transhipped (*cf. J.*, 1921, 322).—(*U.S. Com. Rep.*, Apr. 30, 1921.)

#### GENERAL.

**A New Colour Index.**—The last edition of "Farbstofftabellen" by Dr. Gustav Schultz, published in 1914, has become out of date owing to the great changes in the colour industry brought about by the war, and for some time past those interested in the production and application of dyes have felt the need of a new book dealing concisely with the constitution and properties of the whole range of colouring matters. The Council of the Society of Dyers and Colourists has, therefore, now decided to publish a Colour Index to supply this want, and Dr. F. M. Rowe has been appointed to edit it. In order to ensure accuracy and to include the maximum amount of available information, the Society of Dyers and Colourists has invited colour manufacturers throughout the world to co-operate in this publication by supplying the trade names, constitution, and properties of all known dyes of technical importance, and the proofs of the book will be checked by an expert revising committee representing every section of colour production and use. All references to patent and scientific literature will be brought up to date and checked in order to eliminate the errors which are to be found in previous reference books of this type. The Colour Index will be published in twelve parts, and every effort will be made to issue the first part within six months.

**The Coal-tar Colour Industry in Italy.**—When the manufacture of coal-tar colours in Italy was started in 1915, considerable difficulties were encountered owing to the lack of technical experience and the need for economising fuel and employing Italian raw materials as far as possible. Production commenced with the colours most in request and which could be manufactured most easily, viz., Snlphur, Azo, and triphenylmethane dyes. These three groups constitute roughly 60-70 per cent. of the Italian consumption, which prior to the war amounted to 6500 metric tons per annum (Sulphur dyes 1500 t., Azo dyes 3000 t.). The present output of 1300-1400 t. of Sulphur dyes and 2500 t. of Azo dyes is not so inadequate as has been affirmed. The criticism that fast cotton colours and vat dyes, like indigo and indanthrene, are not produced is held to be unwarranted, as the demand is too small. However, it has been announced that these colours are now to be manufactured by the Italcia and Bonelli companies. At present the industry is passing through a crisis which, apart from the general trade situation, is accentuated by the total lack of protective duties and by the fact that German reparation colours (*cf. J.*, 1920, 4382) are being sold in Italy at prices lower than those of similar goods

imported privately from Germany. If such obstacles were removed the Italian dyestuff industry would make much greater progress.—(*Giorn. Chim. Ind. ed App.*, Apr., 1921.)

**The Camphor Industry.**—The Japanese camphor monopoly and the remarkable growth of celluloid manufacture in Japan, together with the correlated decrease in the exportable surplus of camphor, have stimulated the search for new sources of supply and for substitutes in the manufacture of celluloid. There is little chance of an increased supply from Japan as the hope of obtaining the product by distilling the leaves has been disappointed, and although the camphor from the young trees is said to be unsatisfactory, the maintenance of the Japanese output implies the cutting of trees less than 35 years old, whereas the highest yield is not obtained until the trees are 60 years old. American buyers are looking to China for new supplies, as there is an immense number of camphor trees in the Shan States of South-West Yunnan. In Fukien the remaining trees are more or less inaccessible, but experimental production from wild and planted trees is being carried out in the island of Hainan. A Chinese authority has suggested that a foreign syndicate should start operations in South Hainan and South Kiangsi, and extend them later to Kweichow, Kwangtung, and Kwangsi. In the United States, where camphor production began in 1904, the Celluloid Co. now has 3000 acres planted at Satsuma, the Arlington Co. 12,000 acres at Waller, and planting has also been carried out in Texas and in the San Joaquin Valley, California; the yield approximates 120 to 150 lb. of camphor per acre. As the Philippines offer prospects of successful cultivation, an American company has acquired there the control of a large area of suitable land. The camphor tree has been grown more or less successfully in Madagascar, the Canary Islands, Buenos Ayres, and Algeria, but it is to be feared that the true species *Cinnamomum camphora* is sometimes confused with the less camphoraceous *C. glanduliferum*. Within the British Empire, in addition to the Federated Malay States, Ceylon, and Mauritius, the species grows well in Burma, the Nilgiris, Dehra Dun, at Amami in Tanganyika, and in Queensland; experiments in South Africa suggest that the tree might be profitably cultivated in some of the warmer and moister parts of the Union. It is clear that the camphor tree can be grown successfully within the British Empire, and if care be taken to secure the true species, the small but constant yield of camphor obtained by distilling the leaves would be amply remunerative, even at prices below those now obtaining.

Hitherto synthetic camphor has been unable to compete with the natural product. The so-called "artificial camphor" differs widely from the true camphor and consists of pinene hydrochloride, prepared by treating turpentine oil with dry hydrogen chloride, but true synthetic camphor is made by oxidising borneol or isoborneol prepared from pinene hydrochloride or hydrobromide. The synthetic product closely resembles true camphor, but is usually optically inactive and often contains small amounts of borneol and other substances. As camphor can be manufactured in Formosa at an export price of 6d. per lb., this constitutes the limit to the cost of its production elsewhere. It is stated that 36 gallons of turpentine is required for 99 lb. of camphor, and this, with turpentine at 46s. per cwt., implies a cost of about 1s. 4d. per lb. of camphor for turpentine alone. Efforts should be made to obtain supplies within the British Empire by cultivating the tree in suitable areas and by manufacturing the synthetic product in the United Kingdom (*cf. J.*, 1920, 76, 149, 185, 236, 306, 331, 379, 3812, 1921, 1482).—(*Imp. Inst. Bull.* XVIII, 4).

**A New Process for the Cold Vulcanisation of Rubber.**

G. Bruni has devised a new process for the cold vulcanisation of rubber which is based upon the formation in the nascent state of thiocarbonyl, or other substituted thio-ureas, within the mass of the rubber by the interaction of carbon bisulphide on aniline in presence of zinc oxide or other metallic oxides.—(*G. Chim. Ind. ed App.*, May, 1921.)

**Synthetic Nitrogenous Fertilisers in Germany.**

—The recent addition of 700 million marks to the capitalisation of the chief German dye companies (*cf. J.*, 1921, 174 *n*) promises well for the future of the nitrogen-fixation industry, since all the associated firms have expressed their intention to allocate a considerable proportion of the new capital to the development of the nitrogen works. Prior to the war the annual production of nitrogen was about 100,000 metric tons, derived from gas and coke-oven installations; to-day these undertakings supply only a small proportion of the total output (gas works about 5 per cent.). In a recent review of the subject, Dr. Bueb, managing director of the Nitrogen Syndicate, stated that the annual production of cyanamide was limited to about 100,000 tons (fixed nitrogen), that the maximum annual output capacity of the synthetic ammonia plants was at present about 300,000 t., and that of the gas and coke-oven plants about 100,000 t.; total 500,000 t. of fixed nitrogen. The days of the use of Chile saltpetre in German agriculture are clearly numbered. Whereas in 1913, 770,000 t. was imported, the imports in 1920 were at the rate of only 40,000 t. per annum (Jan.—Oct. 31, 284 t.). This reduced importation meant a clear saving to the German people of over 2000 million marks at the current rate of exchange. It is worthy of note that agriculturists have at last become convinced that sulphate of ammonia is the equal of Chilean nitrate in fertilising value. A good deal of ammonium nitrate is now being used, the difficulties due to its hygroscopic nature having been overcome; but the future seems to lie with synthetic urea, on account of its very high nitrogen content, its harmlessness when applied too heavily, and its good keeping qualities; large-scale experiments with it are now being conducted in Bavaria, and results should be available by the end of the year. Hopes are entertained that heavy dressings of nitrogenous fertilisers will help to revive the fallen sugar-beet industry and bring new life to the pastures. Prof. Neubauer, of Bonn, has shown the remarkable value of such heavy applications: within 4 weeks 65 per cent. of the nitrogen applied was converted into vegetable albumin, and if such results can be achieved on the large scale a pasture will be able to support twice as much livestock as hitherto. In order to promote intensive cultivation with nitrogenous fertilisers, the Nitrogen Syndicate has formed a subsidiary company with a capital of 500 million mk. to advance loans to consumers. Dr. Bueb is not in favour of utilising water-power in the nitrogen industry, except in a few of the cyanamide works, as the supply is small and far too fluctuating.

**The Vegetable Oil Industry in Argentina.**—The production of linseed oil in Argentina is sufficient to meet the requirements of domestic paint manufacturers and those in neighbouring countries, but the chief consuming countries usually prefer to import the seed for extraction in their own mills. Now, however, a Dutch company, the Cia. Sud Americana de Cereales, is erecting an oil mill near Rosario which, it is stated, will be the largest grinding plant in the world. Castor oil for industrial and medicinal use is produced in appreciable quantities, but rapeseed, colewort, and maize oils are only made in small amounts. The supply of cottonseed oil, which is produced on a small scale by most of the existing plants, will probably be augmented

as a result of the increased acreage planted to cotton in the El Chaco territory; a new mill especially adapted for cottonseed-oil production is being built. Cottonseed and groundnut oils are both growing in importance as they are used as substitutes for imported edible oils. Olives have been grown experimentally in the province of La Rioja, but the extension of this crop is not likely to be very rapid. Vertical hydraulic presses are usually employed to express the oil, but extraction with benzine or trichlorethylene is used in a few plants; the mechanical equipment of the mills is said to be good.—(*U.S. Com. Rep.*, Mar. 21, 1921.)

**Cinchona Cultivation in the Philippine Islands.**

According to the Director of the Philippine Bureau of Science, large areas in Luzon and Mindanao Islands are well adapted for cinchona cultivation, judging from the success obtained with a small plantation in Baguio under the supervision of the Bureau of Forestry. The climatic and general conditions in the Philippines are very similar to those in Java and India, where 90 per cent. and 10 per cent. respectively of the world's output are produced.—(*U.S. Com. Rep.*, May 18, 1921.)

**The Italian Mineral Industry.**

—Italy requires normally from 10 to 11 million tons of coal annually, but since the armistice only about 600,000 t. has been imported, largely owing to decreased supplies from Great Britain. Little can be expected from the coal deposits at Heraclea in Asia Minor (*cf. J.*, 1920, 412 *n*), now under Italian control, not only on account of political unrest and lack of transport facilities, but because of the inferior quality of the coal. It appears that Italy will have to depend on imported fuel for some time to come, as the development of hydro-electric power on a large scale is impossible because of the great cost of erecting power stations and transmission lines. Italy produces a negligible amount of mineral oil, and the lignite is of poor quality, although it can be used to a limited extent when briquetted with powdered coal. Sufficient iron ore occurs in the island of Elba and at Cogne in the Val d'Aosta to supply the existing blast furnaces, but the production of pig iron by no means meets the demand; in 1919, 239,710 metric tons of pig iron was produced and 223,811 t. imported. An extension of electric smelting to other ores is feasible, and this would help to free the metallurgical industries from dependence upon imported fuel. Italy is a very important producer of mercury (*cf. J.*, 1920, 204 *n*), especially since the accession of the Idrian mines; certain financial interests have considered the formation of a syndicate to sell the output of the Italian and Spanish mines, thereby ensuring control of the European market. For some time past the Italian sulphur industry has been declining from its former leading position, mainly through competition from the United States, labour difficulties, high costs of production, and inability to improve the output owing to the excessive subdivision of the mines into small holdings. Italy is still the leading producer of pumice, which is obtained chiefly from the volcanic island of Lipari, and nearly all of which is exported to the United States. A large proportion of the output of talc is exported, but of late both production and exports have fallen off. The output of zinc ore, which prior to the war exceeded 150,000 metric tons yearly and was nearly all exported, has fallen off by over 50 per cent. since 1914. Production of zinc metal is very small—1282 t. in 1919—and imports amount to less than 20,000 t. The output of lead ore has declined steadily from 44,654 t. in 1913 to 32,130 t. in 1919, and that of lead metal from 21,674 t. to 16,530 t. There are five producing copper mines, which in 1919 supplied 16,653 t. of ore, compared with 83,302 t. in 1918 and 89,487 t. in 1913; 1243 t. of

metal was produced and 79,425 t. imported. The following table shows that production of aluminium has been steadily increasing but is still insufficient to supply the entire home demand:—

Production.		Imports.		Production.		Imports.	
Metric tons.		Metric tons.		Metric tons.		Metric tons.	
1913	874	483	1917	1,740	3,996		
1914	937	312	1918	1,715	5,008		
1915	904	1,665	1919	1,673	1,602		
1916	1,120	2,132					

The demand for asphalt or asphalt products is not large and the home production, largely concentrated in Sicily, almost satisfies requirements.

Provisional figures for 1920 of the mineral production in Italy, including the new provinces of Trentino and Venezia Giulia, are as follows:—

	Metric tons.
Iron ore and ferro-manganese	423,300
Manganese ore	29,140
Copper ore	6,360
Lead ore	36,325
Zinc ore	98,090
Silver	500
Antimony	125
Iron and copper pyrites	322,450
Mercury	1,325
Fuel:—	
Lignite	1,662,430
Anthracite	23,600
Coal (bituminous)	129,715
Bituminous shale	22,000
Shale (lithological)	495
Sulphur, raw and ground	233,000
Petroleum	4,750
Asphalt and bituminous rock	108,600
Bauxite	37,960
Graphite	4,190

—(U.S. Com. Rep., Mar. 2, 14, 1921.)

## LEGAL INTELLIGENCE.

### ALLEGED INFRINGEMENT OF GERMAN SULPHUR-BLACK DYE PATENT. *A.-G. für Anilin Fabrikation in Berlin v. Levinstein, Ltd.*

With reference to the above case, of which a short report appeared in our last issue (p. 220 B), a correspondent writes that the decision that the use of sodium dinitrophenolate with sodium sulphide and sulphur is a different process from the use of dinitrophenol with sodium sulphide and sulphur will appear surprising to chemists, but the Court was obviously and, in his opinion, rightly impressed by the evidence that the plaintiffs—the patentees—in their own manufacture used sodium dinitrophenolate and kept it as “a factory secret of considerable importance.” Two passages from the judgment are worthy of reproduction:—

“The plaintiffs themselves from the first manufactured their dye by using the sodium salt and not the dinitrophenol; the manufacture started soon after the date of the patent (1900), but when precisely does not appear. We regard this fact as one of very great importance. Dr. Liehmann, who was one of the principal witnesses for the plaintiffs, says that in 1913 he was for the first time told that the black dyes in question could be made from sodium dinitrophenolate, and that the plaintiffs were in fact making their dye in this way. (See evidence given in camera, page 67, question 2807 and following). He obviously regarded this as a matter of considerable importance, and as one which the plaintiffs would desire to keep to themselves.”

Further:—

“We come the more readily to the conclusion that the plaintiffs and their advisers did not know that it was possible to obtain their dye by the substitution of the sodium dinitrophenolate for dinitrophenol because, if they did know it, it is difficult to understand why such possibility was not pointed out in the specification. We think it is clear on

the evidence that for commercial purposes the substitution in question was economically an advantage and accordingly, by omitting to mention it, they were, on the assumption that they knew the facts, laying the patent open to attack on the ground that the patentees had not informed the public of the best way known to them of putting the invention in practice.”

It is important to understand that this is not a decision that when the use of an acid is patented working with the salt is not an infringement. Another point of first-rate importance for chemical patentees is the distinction drawn by the Court between this case and the Half-Watt Lamp case, *The British Thomson-Houston Co., Ltd., v. Corona Lamp Works, Ltd.* (cf. J., 1918, 117 B).

“The remaining objection to the patent is that of insufficiency of description. Practically it resolves itself into this, that the patentees leave undefined the limits within which variation of proportions is permissible. The answer to this objection, we think, is this, that the proportions are not an essential part of the description. The patentee gives an example, by following which the result can be obtained. The possibility of varying the proportions without effecting the result is merely mentioned as one of the points in which the patented process differs from Cassella's. Under such circumstances we see no objection in point of law to a specification which, while giving a definite prescription, leaves the proportions which a particular operator may employ to his own discretion, informed possibly by actual experiment. The case here is not like that of the British Thomson-Houston Company, Ltd. v. Corona Lamp Works, Ltd. (Reports of Patent Cases, 37, p. 227), recently before this Court; there the larger diameter of the filament was of the essence of the invention and, this being so, it was held that the specification was insufficient because the patentee did not state what he regarded as a large diameter. For these reasons we agree with Mr. Justice Sargant that the objections to the validity of the patent fail.”

This part of the judgment, our correspondent thinks, will make it a much-quoted case.

## PARLIAMENTARY NEWS.

### HOUSE OF COMMONS.

#### *British Dyestuffs Corporation, Ltd.*

Answering Sir W. Barton, Sir R. Horne said that the Government holding in the British Dyestuffs Corporation consisted of 850,000 £1 preferred ordinary and 850,000 £1 preferred shares, both fully paid; at current market quotations, these shares would be worth about £580,000, but in view of the present trade depression this figure afforded no adequate indication of the ultimate value of the investment. Recent changes in organisation indicated that whilst substantial progress had been made, the board was not entirely satisfied with the position. The Government directors were in full accord with their colleagues as to the necessity of taking every possible measure to secure greater efficiency in the conduct of the undertaking, and steps had been taken to that end.—(June 9.)

#### *Lead Paints.*

Mr. Hannon asked if the recent experience of the Office of Works had shown that leadless paint were neither as economical in use nor as durable as lead paints, and whether any report had been prepared by H.M. Office of Works on the relative economic value of lead paints and leadless paints. In reply, Lieut.-Col. Sir J. Gilmour stated that

the experience of the Office of Works with leadless paints was being carefully reviewed for the information of the Home Office and in connexion with the general question of the prohibition of the use of lead paints which was to be discussed at the International Labour Conference in October.—(June 13.)

#### Dyestuffs Development Committee.

Capt. Elliott asked the President of the Board of Trade whether an application had been received from the British Association of Chemists, as representing technical chemists engaged in the industries making and using dyestuffs, for representation on the Development Committee to be set up under the Dyestuffs (Import) Regulation Act, 1920. In reply, Mr. Baldwin said that the application had been received, and although the object was to secure a fully representative committee this did not imply the appointment of a representative of each individual association. Satisfactory progress had been made in the constitution of the committee and an announcement would be made at an early date.—(June 20.)

#### Imports from European Countries.

Replying to Capt. Coote, Mr. Baldwin stated that the imports of pig iron and steel from Europe into the United Kingdom during 1920 were valued at £11,853,528 and included:—Pig iron, 177,202 tons; ingots, not special steel, 7427 t.; blooms, billets, slabs, steel, not special steel, 54,480 t.; special steels, 310 t.; the total imports of all descriptions of iron and steel and manufactures thereof were 467,432 t., worth £11,853,528; and the total value of all imports, except food and drink, into the United Kingdom from Germany was £28,319,000.

In an answer to Sir W. Barton, Sir P. Lloyd-Greame said the imports from Germany during the period January—March, 1921, included the following items:—Finished coal-tar dyes, other than alizarin and synthetic indigo, £472,104; crude zinc, £333,925; wire, other than uninsulated electric and fencing wire, £156,278; machines and machinery, not specified, £129,588.—(June 20, 21.)

#### The British Cellulose Co.

Lord H. Cavendish-Bentinck asked the Chancellor of the Exchequer whether he was aware that the British Cellulose Co. had stated in its prospectus that the company's plant was capable of turning out a large quantity of artificial silk, although at the time of issue of the prospectus the promoters of the company had not discovered the process of making an artificial silk which would dye; and if the Government was responsible for the issue of this prospectus. In reply, Mr. Young said that the Chancellor of the Exchequer was advised that the silk produced by the company had always been capable of taking dyes. The Government was not responsible for the issue of the prospectus, but merely approved the reference in it to the Government's acceptance of preference shares.—(June 21.)

#### H. M. Factory Gretna.

Sir L. Worthington-Evans, in reply to Major W. Murray, stated that the Government had decided to dispose of the factory at Gretna.—(June 21.)

#### Duty on Spirits.

Mr. Young, replying to Major Kelley, said that an amendment was moved to the Finance Bill last year to the effect that spirits used in the manufacture of perfumes and similar substances should be treated similarly to spirits used in medicinal preparations or for scientific purposes. The amendment was withdrawn and he saw no reason to modify the decision then taken.—(June 21.)

## REPORTS.

**MINES AND QUARRIES: GENERAL REPORT, WITH STATISTICS, 1920.** By the CHIEF INSPECTOR OF MINES. Pt. I.—DIVISIONAL STATISTICS. Pp. 27. London: H.M. Stationery Office. 1921. Price 3d.

The number of mines operated in 1920 was 2851 under the Coal Mines Act, and 498 under the Metalliferous Mines Act, compared with 2843 and 495, respectively, in the previous year. In addition, there were 5479 quarries at work (5135 in 1919), making a grand total of 8828 mines and quarries, or 355 more than in 1919. The total number of workers employed in or at the mines and quarries was 1,337,297, of which 93·3 per cent. was employed under the Coal Mines Act, compared with 1,270,050 and 93·8 per cent. in the preceding twelve months. There were 1134 deaths from accidents, and the accident death-rate under the Coal Mines Act was 0·88 per 1000, as against 1229 and 0·94 per thousand in 1919. The appended table summarises the output of minerals from all sources in the United Kingdom during 1920:—

*Summary of output of minerals from mines, quarries and brine wells.*

	Tons. 1919.	Tons. 1920.
Alum shale .. .. .	5,539	4,848
Arsenic .. .. .	1,997	2,527
Arsenical pyrites .. .. .	1,178	75
Barium compounds .. .. .	64,150	60,087
Bauxite .. .. .	11,020	9,221
Bog ore .. .. .	2,179	8,045
Chalk .. .. .	3,747,105	2,629,406
Chert, flint, &c. .. .. .	84,303	50,082
Chromite of iron .. .. .	1,100	150
Clays* and shale .. .. .	11,030,418	7,765,965
Coal .. .. .	229,532,081	229,779,517
Copper ore and copper precipitate .. .. .	275	372
Fluor spar .. .. .	54,683	36,860
Gold ore .. .. .	1	—
Gravel and sand .. .. .	2,757,052	2,048,427
Gypsum .. .. .	286,978	220,003
Igneous rocks .. .. .	5,820,091	4,387,703
Iron ore .. .. .	12,707,475	12,254,185
Iron pyrites .. .. .	6,659	7,336
Lead ore .. .. .	15,399	13,868
Lignite .. .. .	150	—
Limestone (other than chalk) .. .. .	11,227,817	9,537,495
Manganese ore .. .. .	12,375	12,078
Natural gas (cb. ft.) .. .. .	(95,000)	(90,000)
Oakum, timber, &c. .. .. .	14,957	10,547
Oil shale .. .. .	2,842,582	2,763,875
Rock salt .. .. .	108,033	90,938
Salt from brine .. .. .	2,060,237	1,817,142
Sandstone .. .. .	2,183,816	1,699,858
Slate .. .. .	215,269	104,098
Soapstone .. .. .	361	688
Sulphate of strontia .. .. .	4,183	1,872
Tin ore (dressed) .. .. .	4,858	5,156
Tungsten ores .. .. .	94	166
Uranium ore .. .. .	60	—
Zinc ore .. .. .	5,064	6,938
Total .. .. .	284,600,799	275,384,528

\* Including china clay, china stone and mica clay.

**REPORT ON THE COMMERCIAL AND INDUSTRIAL SITUATION OF SWEDEN AT THE END OF 1920.** By H. KERSHAW, Commercial Secretary to H.M. Legation, Stockholm. Pp. 40. Department of Overseas Trade. London: H.M. Stationery Office. 1921. Price 1s.

Among the many difficulties with which Swedish industries had to contend during 1920 were severe competition from Germany, high cost of production partly due to the high price of coal, high wages, the 48-hour week, and strikes. Increased protection was solicited by the iron and steel, glass, and certain other industries, but the Government authorities decided against any general rise in customs duties and import prohibitions, although these were recommended for adoption in certain isolated cases. The timber industry was prosperous, and the year

was a very profitable one for paper and pulp manufacturers, the exports comprising 585,000 tons, dry weight, of bleached and unbleached sulphite pulp (561,325 t. in 1919); 141,000 t. of bleached and unbleached sulphate pulp (119,074 t.); and 281,965 t. of mechanical pulp, wet and dry (207,712 t.). By the end of the year large stocks had accumulated, the European demand had fallen off but that from the United States had increased, and generally, prospects were uncertain. Output in the iron and steel industry was considerably below the average of the previous ten years owing, it is suggested, to labour troubles, but the export trade was good. At the date of the report the industry was in a difficult position and clamouring for protection. Exports of iron ore amounted to 3,736,000 metric tons, compared with 2,419,000 t. in 1919 and 6,440,000 t. in 1913 (a record year). The Lapland ore, unlike the ore in Central Sweden, is high in phosphorus, but the supply is practically inexhaustible; it is exported chiefly to Germany. Conditions in the chemical industry were fairly good until the summer, and then became steadily worse; glass manufacture is being hit by Belgian competition, but production in the match industry is gradually returning to the pre-war level. A law has been passed prohibiting the use of yellow phosphorus in the manufacture of matches. The high price of coal has led to serious consideration of the country's water-power resources, the estimated supply of water power during about nine months in the year being 6,750,000 h.p. The total production of electric energy at the State power-stations in 1920 was 740 million kw.-hrs., compared with 685 millions in 1919. Rapid progress has been made in the electrochemical industry, which now exports considerable quantities of calcium carbide, nitrolim, chlorates, perchlorates, sodium sulphate, chloride of lime, and electrometallurgical products such as iron and steel, ferrosilicon, ferromanganese, zinc and lead.

The import and export returns for 1920 include the following items:—

#### Imports.

	Inc. or dec. over	
	1920.	1919.
	Metric tons.	Per cent.
Sugar, all kinds .. .. .	60,425	+540.3
Hides and skins, undressed .. .. .	11,912	- 1.7
Mineral oils .. .. .	189,730	+ 16.7
Coal tar .. .. .	11,367	+ 18.5
Vegetable oils and fats .. .. .	17,690	- 57.4
Linseed .. .. .	27,555	+ 66.1
Oilcake .. .. .	64,356	- 6.2
Vegetable tanning materials, extracts .. .. .	3,589	- 72.3
Zinc white, lithopone, barytes (white) .. .. .	5,311	+102.7
Salt, domestic .. .. .	26,843	+ 87.4
Sodium sulphate and bisulphate .. .. .	50,337	+279.6
Soda and sodium bicarbonate .. .. .	18,763	+ 22.1
Chile saltpetre .. .. .	23,048,584	- 0.7
Calcium nitrate .. .. .	22,302	+301.3
Phosphates, crude .. .. .	112,853	+ 62.8
Stassfurt salts .. .. .	49,508	- 60.4
Pyrites .. .. .	114,900	+ 60.7
Coal .. .. .	2,806,303	+ 45.5
Coke .. .. .	358,259	+ 28.4
Briquettes .. .. .	16,973	+ 2.5
Iron and steel .. .. .	212,878	+ 71.3
Copper, crude .. .. .	18,178	+ 24.2

#### Exports.

Pulp, wood:—		
Mechanical, wet .. .. .	214,210	+ 34.9
Mechanical, dry .. .. .	57,755	+ 14.0
Sulphite cellulose, bleached .. .. .	44,182	+ 54.1
Sulphite cellulose, unbleached, wet .. .. .	40,733	+ 61.1
Sulphite cellulose, unbleached, dry .. .. .	511,859	- 1.9
Sulphate cellulose .. .. .	140,548	+ 18.0
Cardboard, paper, etc. .. .. .	234,999	+ 15.7
Cement .. .. .	68,191	+ 83.2
Superphosphates .. .. .	42,376	+279.7
Matches, wooden .. .. .	36,450	+ 28.2
Iron and steel (includ. manufactures) .. .. .	222,678	+ 8.7

The writer of the report draws attention to the great loss incurred by British manufacturers in carrying on their Swedish business through agencies established in other Scandinavian countries.

## COMPANY NEWS.

### BRUNNER, MOND AND CO., LTD.

In his speech to the annual general meeting, held at Liverpool on June 15, Mr. Roscoe Brunner (chairman of directors) said that the new company—Synthetic Ammonia and Nitrates, Ltd.—had erected at the works of Castner Kellner a semi-large scale plant of the Haber type, but with improvements, and this had been running for some weeks producing about 1 ton of ammonia per day, soon to be increased to 2 tons. The plant, which was the most complicated the company had erected, was designed and started with exceptionally few troubles, and the experience gained would render much easier the designing and operating of the large plant to be erected at Billingham. The technical results obtained confirmed the expectation that with any reasonable cost of construction and any probable selling price, the business would be a profitable one.

Referring to the present industrial depression, Mr. Roscoe Brunner said that trade had suffered the severest slump it had ever known and that recovery must necessarily be slow. The wealth destroyed in the war could only be replaced by hard work and saving, and to save the people must be able to buy in greater quantities at a lesser cost. Costs must be reduced by lowering wages, since in the cost of production of every article wages is by far the largest item, averaging at least 70 per cent. The reductions in wages had not gone nearly far enough. Salaries, too, must be reduced, and the Government must lead the way in economising; every Government service which is not of vital necessity must be scrapped. When the new capital was issued in April, it was intended to continue the erection of the Wallerscote Works and the Synthetic Ammonia Works at Billingham, but owing to the incidence of the coal strike and the uncertainty of the immediate future, the company's building programme had been postponed in its entirety.

The gross trading profit for the year ended March 31 last was £493,165, after deducting depreciation, compared with £826,000 in the previous year, but investment revenue increased from £460,000 to £655,479, and the net profit was £936,000, as against £1,126,000. The decrease in the total dividend on the ordinary shares from 1½ to 8 per cent. is partly due to the increased capitalisation and partly to the severe depression which set in last November.

### ZINC CORPORATION, LTD.

The annual general meeting, held in London on June 15, was addressed by Mr. F. A. Govett, chairman and managing director.

With present working costs, working hours, and metal prices, the company's mine cannot be worked at a profit, and operations are now confined to producing zinc concentrates from the accumulated tailings (664,633 t.) and middlings (28,862 t.), of which the supply should last about 2½ years, and from which the profit at current prices would cover the dividend on the preference shares. No development work is being done and the ore reserves at the close of the year 1920, exclusive of the zinc lode, were estimated at 2,115,700 tons of ore, averaging 14.6 per cent. lead, 2.6 oz. silver, and 9.4 per cent. zinc. The company has subscribed a further £50,000 to the Electrolytic Zinc Co., has invested £15,000 in a combine called the British-Australian Lead Manufacturers, to make subsidiary lead products, and has acquired an interest in another combine—the Broken Hill Globe Timber Co.—to ensure a supply of timber. The prosperity of the



company, said Mr. Govett, depends upon the cost of producing lead and zinc concentrates, and the price of lead and spelter; the former is practically entirely a question of the cost of labour, and the latter mainly one of the world's consumption and demand, which depend upon price and hence upon cost of production, i.e., labour. The cost of labour is thus the vital factor throughout. For 19 months the company's mine at Broken Hill was closed down owing to a strike of labour, and when work was resumed in November on the terms of an award, it was found that those terms made profitable mining impossible, except with lead at an abnormally high price. If the 35-hour week prescribed by the award were abandoned for the 44-hour week, mining could be profitably carried on with lead at a considerably lower price.

The profit for the year ended December 31, 1920, was £34,409, out of which the preference dividend was paid for the first half-year (10 per cent.), but owing to the uncertain outlook no distribution has been made for the second half-year.

## TRADE NOTES.

### BRITISH.

**Nigeria in 1919.**—The year 1919 was marked by a revival in trade owing to the demand for oil-bearing materials, particularly palm oil, palm kernels, and groundnuts. Trade, however, was hampered by the inability of the railway to deal with all the produce. Cotton is quickly regaining its pre-war importance and the cocoa industry is developing steadily, the export in 1919, 25,511 tons, being five times greater than in 1914. Cultivation of rubber and of kola are being encouraged. In the Northern Provinces 88 tin-mining companies worked throughout the year and produced 8168 t. of tin ore (8434 t. in 1918). Gold mining was continued near Minna, in the Nupe Province, and 319 oz. of alluvial gold was won. The output of coal was 137,844 t. compared with 148,214 t. in 1918, the reduction being due to labour difficulties.

The total value of the imports during 1919, exclusive of specie, was £10,798,671, as against £7,423,158 in 1918. The British Empire supplied over 96 per cent. of the total imports, which included:—Kola nuts, 162,701 centials; salt, 54,755 tons; kerosene, 2,069,353 galls; iron, steel, and manufactures of, 9972 t.; soap, 3016 t. Exports were valued at £14,675,789, exclusive of specie (£9,511,971 in 1918); they included:—Kennebec, 57,074 t.; rubber, 892,081 lb.; shea products, 1729 t.; tin ore, 7685 t.; palm oil 90,967 t.; palm kernels, 216,913 t.; groundnuts, 39,334 t.; hides and skins, £1,262,140. Eighty-five per cent. of the exports was directed to the British Empire, but both import trade and export trade with the United States have increased.—*Col. Rep.-Ann., No. 1064, Mar., 1921.*

### FOREIGN.

**Foreign Company News.**—**Czechoslovakia.**—The following companies have recently been formed for the manufacture of artificial silk:—Gebrüder Kader, Mährisch-Ostrau, capital 20 million mk., with a daily output capacity of 1000 kg.; Elberfelder Glanzstoff-fabriken, Aussig, capital 50 million mk.; Th. Liebig, Reichenberg, capital 20 million mk.; Wolf and Schleim, Jos and Löwenstein and the Zivno Bank, Theresienthal near Arnau, capital 10 million mk.—(*Chem. Ind., May 30, 1921.*)

**France.**—The Etablissements Kuhlmann is paying dividend of 12 per cent., the same as last year; the carry forward is 992,496 fr.

The Société d'Electro-Chimie et d'Electro-Metallurgie announces a net profit of 3,709,493 fr., and a dividend of 45 fr.

A net profit of 17,323,657 fr. was made by the Société Saint-Gobain in 1920; 7 million fr. was carried to reserve and the dividend payable is 245 fr. per share (210 fr. for 1919).—(*Rev. Prod. Chim., June 15, 1921.*)

**Germany.**—Kalle and Co., A.-G., Biebrich, made a net profit of 3,030,000 mk. in 1920 and is paying 20 per cent. The dyestuffs produced by the company fetched good prices and the pharmaceutical section developed satisfactorily, but during the second half of the year sales decreased owing to foreign competition and the industrial crisis.

The net profit of the Vereinigte Kunstseidefabriken, A.-G., Frankfurt, for 1920 was 270,426 mk., the dividend is 9 per cent., and the capital is being raised to 8 million mk. The manufacture of staple fibre was found to be unremunerative and that of artificial silk was resumed in the middle of the year.

A dividend of 25 per cent., plus a bonus of 500 mk., making a total of 75 per cent., is to be distributed by the A.-G. für Chemische Industrie, Gelsenkirchen; 1,140,000 mk. was written off and 2 million mk. carried to maintenance account, leaving a net profit of 5,800,000 mk.

The turnover of the Chemische Fabrik auf Aktien vorm. E. Schering, Berlin, increased considerably, but early in the year production did not meet demand owing to lack of fuel and other difficulties. The net profit was 3,592,381 mk. and the total dividend 25 per cent.; the capital is to be raised to 11 million mk. to extend and modernise the works at Eberswalde.

The output of the Chemische Fabriken vorm. Weiler-ter-Meer, Uerdingen, in 1920 was less than one-half of the pre-war average, but owing to good prices in the first months the results for the year were favourable. Wages and salaries rose from 5,560,000 mk. in 1919 to 18,100,000 mk. in 1920, the number of employees remaining about the same. The balance to the credit of profit and loss was 7,133,367 mk. and dividends amounting to 185 per cent. have been paid or are payable.

Other dividend announcements are as follows:—Köln Rottweil A.-G., 16 per cent. (capital 66 mill. mk., to be raised to 100 mill. mk.); Dynamit A.-G. vorm. Alfred Nobel and Co., 16 per cent. (capital 72 mill. mk., to be raised to 100 mill. mk.); Rheinische-Westfälische Sprengstoff A.-G., 12.8 per cent. (capital 26 mill. mk., to be raised to 40 mill. mk.); Siegener Dynamit Fabrik, 12.8 per cent. (capital 1.2 mill. mk., to be raised to 1.8 mill. mk.). The Farbenfabriken vorm. F. Bayer and Co., Leverkusen, is paying 20 per cent. for the year and the capital is to be raised to 430 million mk.—(*Chem. Ind., May 23, 30, 1921.*)

**Poland.**—A new company, the Polish Oil Co. "Polpetrol" has been formed with a capital of 60 million Polish marks. With the help of British and French capital it has acquired 490 acres of oilfields in the Sanok district, where eight wells have been sunk, and is about to purchase an additional 1162 acres upon which six shafts will be constructed during this year. The "Galician Joint-stock Oil Industry Co." is another new oil company, with a subscribed capital of 16,250,000 mk (Polish mk.=1s. at par, now about 0.04d.).—(*U.S. Com. Rep., Apr. 15, 1921.*)

**Spain.**—The Industrial Resinera Ruth company has been formed by a German firm and the Sociedad Union Resinera Española, the latter subscribing 70 per cent. of the capital. The company will manufacture enamels, varnishes, paints, glue, aniline, and other chemical products, including synthetic camphor.—(*U.S. Com. Rep., Apr. 15, 1921.*)

**Yugoslavia.**—The Kastel Factory of Chemical and Pharmaceutical Products, recently founded at Karlovatz, Croatia, will be the first company in the Balkans to manufacture medicinal products such as compressed tablets, serums, and medicinal plant products. The company will cultivate medicinal plants on a large property adjacent to the railway between Karlovatz and Ljubljana (Laibach).—(*U.S. Com. Rep.*, Apr. 13, 1921.)

## OFFICIAL TRADE INTELLIGENCE.

(From the Board of Trade Journal for June 9 and 16.)

### OPENINGS FOR BRITISH TRADE.

The following inquiries have been received at the Department of Overseas Trade (Development and Intelligence), 35, Old Queen Street, London, S.W.1, from firms, agents, or individuals who desire to represent U.K. manufacturers or exporters of the goods specified. British firms may obtain the names and addresses of the persons or firms referred to by applying to the Department and quoting the specific reference number.

Locality of firm or agent.	Materials.	Reference number.
Bermuda .. ..	China, glassware .. ..	716
British West Indies ..	Soap, perfumery .. ..	717
.. ..	China, glassware .. ..	718
South Africa .. ..	Crockery, glassware .. ..	705
.. ..	Crockery, glassware, bottles, sheet glass, plate glass .. ..	707
Hungary .. ..	Raw materials for medicines, vegetable fibres, cork .. ..	729
Italy .. ..	Malt .. ..	711
Sweden .. ..	Leather belting .. ..	733
.. ..	Chemicals for pulp, paper and other industries .. ..	734
Morocco .. ..	Galvanised sheets, electric light bulbs (120 volts) .. ..	713
Argentina .. ..	Heavy and fine chemicals .. ..	714

### TARIFF. CUSTOMS. EXCISE.

**Czechoslovakia.**—The "Manipulation" fees on exports affect cardboard, chemicals (except hydrochloric acid), chloroform, cellulose pulp, collodion, cyanide, dyes, faience, glass, gold, kaolin, artificial leather, matches, mineral wax, paper, photographic goods (except films), polish, platinum, porcelain, rubber goods, silver, tar and derivatives thereof, yeast, etc. Such fees are payable on imported hops, leather, photographic films and papers, stoneware, and tannin.

**France.**—Extracted fats (wool grease, etc.) may now be exported without special authorisation.

The export duties on edible and other oilcakes have been abolished. The export of raw cattle bones is permitted subject to a duty of 3 fr. per 100 kg. The export of natural phosphate of lime is allowed until further notice to all destinations.

**Germany.**—Albumin may be exported without licence as from May 23.

**Germany (Occupied Territory).**—Import licences are not required for fats, hides, and iron ore, nor export licences for bricks and certain leather goods.

**Greece.**—A copy of the decree controlling the trade in opium, morphine, cocaine, heroin and preparations thereof may be seen at the Department, 18, Queen Anne's Gate, S.W.1.

The import of sodium carbonate is again allowed. **Hungary.**—For the purpose of the payment of customs surcharge, goods are divided into three

classes. Goods in list A include cardboard, newsprint paper, and certain chemicals, and are surcharged 1100 per cent. List B, on which the surcharge is 1900 per cent., includes paper, fine cardboard, rubber, tanned skins, glassware, pottery, firebricks, crucibles, and certain chemicals. Goods in list C are surcharged 3400 per cent. Full particulars may be obtained on application to the Department.

**Italy.**—As from April 1 the payment of customs duties on a gold basis has been resumed. Payments may be made by certificates to be obtained from the Banks of issue at a price fixed fortnightly by the Minister of the Treasury.

The import of dyes and intermediates is prohibited, but certain exceptions may be specified from time to time by the Minister of Finance.

**Japan.**—The new tariff amendments came into force on June 1.

**Nigeria.**—As from June 1, in order to import morphine, cocaine, ecgonine, diamorphine, and medicinal opium the importer must furnish the Government with a statement in quadruplicate of his requirements and a guarantee that the consignment is required for legitimate scientific and medicinal purposes; the name of the firms supplying the drugs should also be stated.

**Nyasaland.**—The customs tariff has been revised with effect from April 1. All goods (with some exceptions) pay an import duty of 10 per cent. *ad valorem*. Among the articles that are duty-free are manures, disinfectants, insecticides, and drugs of the British Pharmacopoeia.

Export duties are payable on gold and beeswax.

**Poland.**—The import of vinegar is prohibited. Zinc may be exported without licence as from April 16.

**Portugal.**—Export licences are required for, *inter alia*, butter, lard, olive oil, and sugar.

Goods on which export surtaxes are payable include lard, butter, matches, scrap iron, rosin, certain hides and skins, wolfram ore, and tin.

**Rumania.**—The export taxes on certain petroleum products have been modified.

Commission charges are levied on the export of certain seeds, sunflower oil, oilcakes, timber, carbide, carbon bisulphide, bleaching powder, gypsum, soap, salt, bitumen, grease, tar, pitch, petroleum products, lard, cement, tarred paper, old iron, manganese iron, oak bark, and shoemakers's glue.

**San Salvador.**—The import duty on gasoline has been reduced to 1½ centavos (gold) per kg.

**St. Vincent.**—A copy of the regulations relating to certificates of origin under the British preferential tariff may be seen at the Department.

**Spain.**—Modifications of the new provisional customs tariff affect coal-tar colours, aniline oil and hydrochloride, and pulp for paper making.

Surtaxes have been levied on all classes of goods. Within the 8 months ending February 3, 1922, 25,000 metric tons of sugar may be exported.

**Sweden.**—Export prohibitions have been withdrawn from vegetable fibres, premier jus, oleomargarine, malt, starch, flax seed, rape seed, liquorice, and sweetened condensed milk.

Increased import duties are payable on hops, liquorice, cumin, certain spices, vanilla, saffron, incandescent mantles, and wares of gold, silver, and platinum.

**Switzerland.**—Import licences are no longer required for paper, cardboard, bottles, and certain manufactures of iron, copper, and of copper alloys when imported over the Franco-Swiss or Italo-Swiss frontiers.

**Tunis.**—The prohibition of the import of paper on reels and pulp for newsprint paper has been abolished.

**Venezuela.**—The regulations affecting the import, manufacture, and sale of foodstuffs may be seen at the Department.

## REVIEWS.

**FAMOUS CHEMISTS: THE MEN AND THEIR WORK.**  
By SIR WILLIAM TILDEN. Pp. 296. (London:  
George Routledge and Sons, Ltd.; New York:  
E. P. Dutton and Co. 1921.) Price 12s. 6d. net.

Sir William Tilden has written a book which will be not only a very convenient book of reference for chemists, but will appeal to general readers who desire to learn the main characteristics and achievements of some of the great men—from Boyle to Ramsay—who have built up the science of chemistry.

The question at once arises—who shall be included in such a book? As a guide to selection the author has taken the evolution of the atomic theory, and has chosen the men who, in his judgment, have taken the leading part in establishing, step by step, the idea of the chemical element and the quantitative laws of chemical combination, the men who have isolated new elements and those who have explained the structure of molecules from a study of the properties of atoms. If twenty-one names are to be selected from those whose work appeared indispensable to the progress of the theory, chemists could not differ greatly from the author in their voices. In making an independent selection the writer was surprised to find how many of the same names were included; indeed, in the case of the older chemists, he can congratulate himself in "spotting" eleven out of the first twelve. This concordance no doubt is due to the perspective of time: when details are lost the great realities can be seen.

For all the chemists he has chosen Sir William Tilden has given good reason why he has selected them as "indispensables," and why their fame should be preserved; and if we have a doubt in any particular case and think, for instance, that Proust could hardly have persuaded the world that Berthollet was wrong in questioning the universality of "definite proportions," had it not been for Dalton's atomic theory and the law of multiple proportions which Proust himself failed to see—well, we all admit that Proust did brave work and are glad to have the details of his life. Among modern atomic chemists we welcome the inclusion of Alexander Williamson as one of the great upholders of the theory, the inventor of the water-type and the first chemist to introduce the idea of intermolecular exchange, which lies at the root of the modern doctrine of ionisation.

If, where there is so much to praise, it is the critic's business to seek a point or two to debate, one point shall be on a matter of judgment, the other on a small matter of fact. In Dalton's life no mention is made of his experimental determination of the law that all gases under the same conditions expand alike for equal increments of temperature. Under Gay Lussac we find this reference:—"... like Dalton, he also adopted the erroneous idea that the expansibility of all gases was the same." Now Dalton, working with oxygen, hydrogen, nitric oxide and carbon dioxide, found that the actual expansion became less as the temperature rose, but all the gases behaved alike, and it is the equality of expansion of the different gases which he lays stress upon. Surely this generalisation which all continental writers ascribe to Gay Lussac, and was first enunciated by Dalton, had an important influence on the development of the atomic theory—in spite of the fact that neither experimenter determined the true coefficient of expansion. As to the matter of fact—a small fact admittedly—is Sir William Tilden (in company with most other chemists) justified in questioning the authenticity of "the pear-shaped vessel commonly called Cavendish's Endiometer"? The writer has

in his possession a glass "pear-shaped vessel" made by Newman; it was given to him 40 years ago by the Rev. T. H. T. Hopkins, Fellow of Magdalen, with this history:—It was made originally by Newman for Cavendish, and was brought back by Newman after Cavendish's death; it was purchased from Newman as Cavendish's instrument by Professor Daubeny, of Oxford, who left it to his friend Hopkins. The "Endiometer" is a very thick-walled explosion vessel, the platinum firing-wires being fastened by cement in holes bored through the glass-stopper, which is clamped down just as shown in the Cavendish Society's plate.

Every chemist will find some new facts in this book, and will be pleased to see the old facts so skilfully arranged. Sir William Tilden has made excellent use of his materials, and every picture leaves a brilliant impression on the mind: he brings out the environment in which his heroes worked, and the stimulus they gave to their contemporaries. The word "heroes" is used designedly; for the author, if he errs at all, errs by way of admiration, and, only touching lightly on the controversial, rightly sets forth the good that is to be remembered.

HAROLD B. DIXON.

**LES VERNIS.** By CH. COFFIGNIER. Preface by M. HALLER. *Encyclopédie de Chimie Industrielle.* Pp. xviii. + 640. (Paris: J. B. Baillière et Fils. 1921.) Price 40 francs.

Coffignier's book forms a welcome addition to the literature of the varnish industry. It opens with a very full and valuable account of the various gum resins, giving their origin, properties, chemical composition, and where possible tests for their recognition. The oil-resins, balsams, and also the artificial aldehyde-condensation-products, such as Baekelite, now being introduced as substitutes, are described. Asphalt and colouring matters, such as turmeric, aloes, and coal-tar colours follow. The analysis of gums is then given, including the acid and saponification values, which serve as useful guides. The iodine number, which the author rightly carries out with Hubl's solution, and not by Wijs or Hanus, as the gums are more soluble in alcohol than in acetic acid, proves of great value in determining the class of gum; but scant attention is paid to the action of air on the gum resins, as in the weathering of gums.

In the chapters on oils, linseed oil is first treated fairly well, but too little attention is paid to the constants of different varieties, such as iodine values. The same criticism applies to the other oils, such as tung, lumbang, soya, and some of the lesser known oils which are mentioned. This fault, however, is almost remedied in a later chapter on the analysis of oils. Apropos of the iodine value, the author still gives the view that the iodine is absorbed from the various solutions in the form of hypiodous acid, which was disproved years ago (cf. J., 1902, 587). Little mention is found of the important hexabromide test, whilst the obsolete Livache test, and other more or less unknown methods, occupy too much space. The description of turpentine and its substitutes, their preparation, composition and properties are adequately treated.

The cleansing, garbling, crushing and, later, the running of gums, both at the ordinary pressure and in the autoclave, are ably described, and the boiling of the oil to be subsequently added is given, together with numerous recipes for special varnishes. A discussion of the action of driers on oils follows the latest theories with regard to their action on raw oil, but one is surprised to find reference to zinc salts, as, if pure, they have no drying action. No mention is made of the work of Morrell (cf. J., 1915, 108) on the polymerisation of unsaturated oils under

the action of heat, or the bearing of this on the character of the film left on drying, nor is there any reference to the greater permanency of a polymerised ("polylin") film compared with that of an oxidised ("linoxyn") one, and the relation of "super-oxidised" oil to this matter (*cf.* Ingle and Woodmansey, J., 1919, 101).

The volume is very comprehensive, but it leaves an impression that the author has omitted some useful work published in English journals of late years. Nevertheless, the book covers the subject more fully than any English or German work known to the writer, and the only adverse criticism to be made is that the arrangement of the subject matter causes the details to be diffused, instead of being confined under their particular subject titles.

HARRY INGLE.

AN INTRODUCTION TO THE CHEMISTRY OF PLANT PRODUCTS. VOL. I. ON THE NATURE AND SIGNIFICANCE OF THE COMMONER ORGANIC COMPOUNDS OF PLANTS. By PAUL HAAS and T. G. HILL. Third edition. Pp. xiii.+414. (London and New York: Longmans, Green and Co. 1921.) Price 16s. net.

In the preface to the first edition of this book the authors explained that it was designed to meet the needs of students of vegetable physiology, whose chemical knowledge is frequently "deficient in just those branches of chemistry which are of particular importance to the botanist, which is no doubt largely due to the fact that those compounds, which are of interest to the botanist, do not necessarily fit into the scheme of instruction of the chemist." Though nearly nine years have elapsed since then, this statement remains true, and the third edition of the book is on much the same lines as the first, with the exception that certain sections have been added to or rewritten, and that it is now issued in two volumes. Volume I is similar in scope to the full work in the two earlier editions; Volume II, which is to be published subsequently, will deal with physiological problems more fully than has been attempted hitherto.

The fact that the book is now in its third edition shows that it is meeting a real need on the part of students. The subject matter is arranged roughly into groups of products, *e.g.*, fats, carbohydrates, glucosides, tannins, pigments, nitrogen bases, etc. Hydrocarbons are omitted in spite of the fact that plant constituents include such an interesting and varied collection of substances of this class as paraffins, terpenes, rubber and gutta-percha. Similarly resins and essential oils are excluded.

The contents of the various sections seem quite adequate and up-to-date, so far as scientific information is concerned. It is doubtful whether it is worth while to include quite so much elementary chemistry as is given in the section on fats, oils and waxes. No one could read this book with profit without a considerable knowledge of chemistry, and for such readers it is surely unnecessary to explain what is meant by a paraffin and to set out in full the constitutional formulæ for ethane and propane (p. 9). Further, to rise from this to the complex Windaus formula for cholesterol (p. 17) in nine pages argues a greater absorptive capacity and more aptitude for the niceties of constitutional formulæ than the average beginner possesses. Much of the analytical detail concerning oils and fats could also be omitted as it is readily available elsewhere. In dealing with tannins no attempt is made to describe the estimation of tannin, and the section gains by the omission.

To readers of this journal there is no need to enlarge on the commercial and industrial importance of plant materials—the existence of such industries as those dependent on rubber, oilseeds,

foodstuffs, fibres and cotton is an ever-present reminder of the great interests concerned in them. The authors exhibit a laudable desire to emphasise the economic side of the subject by frequent references to the industrial uses of the products they describe and to the processes employed in their preparation. Some of this information needs more thorough revision than it has received. For instance, though it is admittedly difficult to give a crisp, complete and accurate description of a varnish, one of the things it probably never is, is a "mixture of boiled oil with gum resins and oil of turpentine," if the term "gum resins" is used accurately.

The subsection on the "Industrial Uses of Vegetable Fats and Oils" omits all reference to palm-kernel oil except as a soap material, says nothing about the use of cottonseed oil for edible purposes, and states that palm oil has a faint odour resembling that of violets. In making the latter assertion the authors are in good company, but the statement is usually made with the qualification that the oil has this odour when fresh, and as few Europeans have the chance of smelling it in this condition the assertion in this form is more or less credible, but one would hardly associate violets with ordinary palm oil as it reaches this country.

Most of these remarks, it will be seen, refer to the chapter on oils and fats which, however, it is only fair to say, is the weakest portion of the volume. Taken as a whole the book fulfils the objects the authors had in view in writing it, and is in most respects a useful summary of well-selected information regarding those constituents of plants that are of primary importance to the physiologist and the biochemist.

T. A. HENRY.

## PUBLICATIONS RECEIVED.

- A DICTIONARY OF CHEMICAL SOLUBILITIES. INORGANIC. By A. M. COMEY. Second edition, revised and enlarged by A. M. COMEY and D. A. HAHN. Pp. 1141. (New York: The Macmillan Co. 1921.) Price 72s.
- A TEXT-BOOK OF INORGANIC CHEMISTRY. VOL. IX. PART II. IRON AND ITS COMPOUNDS. By J. NEWTON FRIEND. Pp. 265. (London: Charles Griffin and Co., Ltd. 1921.) Price 18s.
- A TEXT-BOOK OF ASSAYING. By C. and J. J. BERINGER. Revised by H. R. BERINGER. Fifteenth edition, revised. Pp. 471. (London: Charles Griffin and Co., Ltd. 1921.) Price 12s. 6d.
- A DICTIONARY OF APPLIED CHEMISTRY. VOL. II. CALCULI-EXPLOSION. By SIR E. THORPE, assisted by eminent contributors. Revised and enlarged edition. Pp. 717. (London: Longmans, Green and Co. 1921.) Price 60s.
- CONDENSED DESCRIPTION OF THE MANUFACTURE OF BEET SUGAR. By F. MURKE. Pp. 175. (New York: John Wiley and Sons, Inc.; London: Chapman and Hall, Ltd. 1921.) Price 15s.
- THE COMPOSITION AND UTILISATION OF EXHAUSTED MOLASSES IN MAURITIUS. By H. A. TEMPART and C. D. D'AVIONE. Bulletin No. 21. General Series. Department of Agriculture, Mauritius. Pp. 15. (Port Louis: F. S. Passingham. 1921.)
- REPORT ON LAC AND SHELLAC. By H. A. F. LINDSAY and C. M. HARLOW. Indian Forest Records. Vol. VIII, Part I. Pp. 162. (Allahabad: The Pioneer Press. 1921.) Price Rs. 2-12-0.

## Manchester Section.

Meeting held at the Grand Hotel on Friday,  
November 5, 1920.

MR. JOHN ALLAN IN THE CHAIR.

### THE INORGANIC CONSTITUENTS OF COAL WITH SPECIAL REFERENCE TO LANCA- SHIRE SEAMS.

BY F. S. SINNATT, M.B.E., M.Sc.(TECH.), F.I.C.,  
M.I.M.I.E.; A. GROUNDS, B.Sc.(TECH.), A.I.C.; AND  
F. BAYLEY, M.Sc.(TECH.), A.I.C.

This paper describes a stage in the study of the inorganic constituents of certain typical seams in the Lancashire coalfield, and records a part of an investigation of the various coals from a number of viewpoints. The work has been carried out under the auspices of the Lancashire and Cheshire Coal Research Association.

It is well known that the coals in the Lancashire coalfield contain, as a rule, a low percentage of inherent ash. Of twenty distinct seams examined recently, none contained more than 6% of ash, whilst a great number contained less than 3%. Bickershaw Yard coal contained as little as 0.8%. The coals vary in properties from those which are practically non-caking to one (Mountain Mine) which possesses probably the highest caking power of any known coal, and yields a perfect metallurgical coke.

In distinguishing the coals the names in most common use have been adopted, but it must be recognised that other terms may be applied to the same seams in different localities.

For the purposes of this paper only the natural inorganic constituents of the seams have been considered. Every care was taken in obtaining the samples on which the work was carried out. A solid pillar was cut from the whole seam the cross-section of which was from 6 to 18 ins., according to the ease with which the coal could be handled. The piece of coal was packed in a box, and carried to the laboratory for examination.

The present investigation included the following objects:—

To determine if any relationship could be established between the composition of the white partings (ankerites) and the ash produced when the coal is incinerated.

To attempt to trace the source of the carbon dioxide evolved when coal is treated with mineral acids.

To investigate the form of combination in which iron exists in the coal substance.

The last subject is of interest in view of the fact that according to the work of Powell and Parr (Bulletin 111, University of Illinois) the greater proportion of the iron present in certain American coals occurs in the form of pyrites, and with this fact is associated the assumption that the iron does not enter into the preliminary stages of the oxidation of coal.

It would appear from the experiments described in the present paper that a proportion of the iron in Lancashire coals exists in the ferrous state, either in the white partings (or ankerites) or some other form of combination.

Most coal seams contain a proportion of white inorganic partings, of which no study has appeared in the literature. It will be seen later that the material may be considered either as substituted calcium carbonate, or as dolomitised siderite. The term ankerite, which is the one generally accepted

in mineralogy for compounds having a similar constitution, has been adopted to distinguish the substance of the white partings.

The ankerites occur in the form of sheets varying in thickness from  $\frac{1}{4}$  in. to a mere film, at right angles to the bedding plane and on the face of the coal, but also frequently on the end of the coal. The layer of material may be so thin as to be transparent, and its presence can only be detected by the fact that after being allowed to stand in the air the coal becomes covered with an opalescent film, consisting of oxidised ankerite. When ankerite occurs adjacent to a band of vitrain it is frequently perfectly white in colour, whilst portions in contact with clarain or durain are comparatively dark, and impregnated with fine coal. It has been observed that a band of ankerite frequently terminates at a point where a layer of fusain occurs. The latter material is highly porous in nature, and it would appear that the ankerites have been deposited from a liquid medium which has travelled horizontally along the layer of fusain.

The ankerites form a distinct line of weakness in the coal, and if the latter is treated with dilute mineral acids, marked disintegration of the lumps is produced, owing to the decomposition of the compounds. The whole of the specimens examined contained varying percentages of iron practically entirely in the ferrous condition, and it will be seen from the analyses that certain examples contained a distinct percentage of manganese.

Average specimens of the ankerites from a number of seams have been analysed, and the results are given in the following table:—

TABLE I.  
Percentage composition of the white partings  
(ankerites).

	Lower Mountain Mine.	Arley Mine.	Ravine Mine.	Sap- ling Mine.	Hoo- ling Con- nel.	Rushy Park.	King Mine.
Calcium oxide ..	27.40	28.56	29.04	42.79	30.08	28.76	30.84
Magnesium oxide ..	13.33	11.51	16.15	0.41	11.18	8.74	5.76
Ferrous oxide ..	14.56	9.81	4.80	13.07	14.16	18.23	18.42
Manganese oxide ..	—	0.82	1.11	—	—	0.59	0.31
Carbon dioxide ..	45.21	41.52	44.33	41.88	44.53	42.46	42.03
Silica ..	—	6.05	2.25	1.70	—	2.45	—
Ferric oxide ..	—	0.60	0.25	0.17	—	0.63	—
Pyrites ..	—	1.11	—	—	—	0.09	2.47
Equivalent to:							
Calcium carbon- ate ..	48.93	51.00	53.14	76.41	53.70	51.36	55.08
Magnesium carbon- ate ..	28.30	24.07	33.65	0.66	23.48	18.28	12.10
Ferrous carbon- ate ..	23.16	15.81	8.40	21.68	22.82	26.18	29.68
Manganese carbon- ate ..	—	1.33	1.80	—	—	0.96	0.50
Calcium sulphate ..	—	—	0.44	—	—	—	—
Silica ..	—	6.05	2.25	1.70	—	2.45	—
Ferric oxide ..	—	0.60	0.25	0.17	—	0.63	—
Pyrites ..	—	1.11	—	—	—	0.09	2.47

Specimens of ankerite obtained from the different layers in certain seams differ considerably in composition; the following analyses show the greatest variation yet encountered:—

TABLE II.  
Ankerite from seam 8ft. thick.

	Top 15 in.	Bottom 7 in.
Silica ..	0.91	2.25
Calcium carbonate ..	52.09	53.14
Magnesium carbonate ..	29.74	33.85
Ferrous carbonate ..	16.15	8.40
Manganese carbonate ..	1.41	1.80
Ferric oxide ..	0.33	0.25
Calcium sulphate ..	nil	0.44

The compounds undergo oxidation on exposure to air and become covered with reddish-coloured ferric compounds.

The following analyses show the change in composition which occurred when a specimen of ankerite was exposed to the air in contact with the coal. It should be pointed out that the two specimens of ankerite were of necessity obtained at a slight distance from one another and some small difference in chemical composition was detected:—

TABLE III.

	Sample freshly mined. Colour: white.	Sample after exposure to air for 12 weeks. Colour: red.
	%	%
Ferric iron .. .. .	0.33	0.91
Silica .. .. .	0.91	0.17
Ferrous oxide .. .. .	9.29	10.58
Manganese oxide .. .. .	0.87	1.08
Calcium oxide .. .. .	29.17	29.31
Magnesium oxide .. .. .	14.22	13.32
Carbon dioxide .. .. .	44.67	44.74

The oxidation of the ankerites is influenced by the presence of carbon dioxide and water. As would be expected, the compounds are freely soluble in water in the presence of carbon dioxide, and the solution which results rapidly undergoes oxidation in the presence of air, with the precipitation of basic ferric compounds.

No accurate method of determining the percentage of ankerites in coal has been elaborated, but an approximate value was obtained for one sample of coal by crushing about 1000 g. until it would pass through a sieve of 4's mesh, and separating all the ankerite that was visible by picking it out with forceps. The coal contained 4.2% of total ash and 3.0% of ankerite. The equivalent weight of ignited ankerite was 1.7%, i.e., 40.5% of the ash was derived from the ankerite present.

Samples of the coals from which the ankerites described above were obtained were incinerated at a temperature of 900° C. and the resulting ashes were analysed with the following results:—

TABLE IV.  
*Composition of coal ashes.*

	Lower Mountain Mine.	Arley Mine.	Ravine Mine.	Sapling Mine.	Hoo Cannel.	Rushy Park.	King Mine.
	%	%	%	%	%	%	%
Silica .. .. .	40.20	43.21	35.00	38.92	32.98	25.49	29.45
Ferric oxide .. .. .	25.66	12.38	9.99	48.40	23.34	38.80	26.32
Alumina .. .. .	25.41	28.47	31.58	3.68	26.86	20.61	29.53
Calcium oxide .. .. .	3.52	7.12	11.63	5.04	6.88	7.08	5.96
Magnesium oxide .. .. .	1.98	2.36	2.45	0.50	3.10	3.22	0.11
Sulphur trioxide .. .. .	2.02	4.30	7.68	0.16	3.04	1.25	1.98
Alkalis and loss .. .. .	1.21	2.16	1.67	3.30	3.80	3.55	6.65
Percentage of ash in the coal	3.00	3.20	5.20	4.00	24.4	12.4	4.6

A comparison of the above results shows that the proportion of the various constituents occurring in the ash is not by any means parallel with that found in the ankerites.

The percentage of carbon dioxide evolved when the coals were treated with mineral acids was determined by a method described in Bulletin No. 7 of the Lancashire and Cheshire Coal Research Association, from which the following results are extracted:—Mountain Mine, 0.57% CO<sub>2</sub>; Arley, 0.18%; Ravine, 0.32%; abnormal sample I., 6.85%; Pemberton 2 ft., 0.72%; Garswood 9 ft., 0.44%; Hoo cannel, 1.84%; Rushy Park, 0.76%; Lower King, 0.77%; Bickershaw Yard, 0.40%.

It was thought that it might be possible to calculate approximately the proportion of the ankerite in a particular seam by the above determination, but it was found that the percentage of carbon dioxide evolved was in excess of that required to combine with the whole of the bases occurring in the coal as ankerites.

The following example shows the result obtained if the carbon dioxide evolved is assumed to be derived solely from ankerites:—

TABLE V.

1. Lower Mountain Mine.  
Ash=3.03%. Carbon dioxide=0.57%.

Constituents.	Coal ash original analysis (100 g.).	Gross constituents of coal ash in 100 g. of coal.	Ankerite analysis.	Ignited ankerite equivalent to CO <sub>2</sub> in 100 g. of coal. g.
Silica .. .. .	40.20	1.218	—	—
Ferrous oxide .. .. .	nil	—	14.56	—
Ferric oxide .. .. .	25.66	0.779	—	0.314
Alumina .. .. .	25.41	0.770	—	—
Manganese oxide (MnO) .. .. .	—	—	—	—
Calcium oxide .. .. .	3.52	0.106	27.40	0.533
Magnesium oxide .. .. .	1.98	0.059	13.33	0.263
Carbon dioxide .. .. .	—	—	45.2	—
Sulphur trioxide .. .. .	2.02	0.061	—	—
Pyrites .. .. .	—	—	—	—
Alkalis and loss .. .. .	1.21	0.036	—	—

From the above consideration it will be clear that the amount of carbon dioxide evolved when coal is treated with mineral acids is more than sufficient to combine with the bases found in the inorganic constituents of the coal, and must be derived from other sources than ankerites.

The source of the carbon dioxide has not been traced accurately, but it will be shown later that a portion of the iron in coal probably occurs as ferrous carbonate, as distinct from that occurring in the ankerites. The percentage of carbon dioxide is, however, of some interest from an analytical point of view. Unless the amount of carbon occurring as carbon dioxide is deducted from that found by combustion of the coal the percentage of organic carbon will be too high. The percentage of carbon dioxide should be deducted from the percentage of volatile organic matter, as presumably the whole of the carbon dioxide is evolved at a temperature of about 900° C. In the case of the abnormal sample, the volatile organic matter determined was too high by 6.8%.

Mr. N. Simpkin, in collaboration with one of us, is continuing certain phases of the work, and has treated the coals with dilute hydrochloric acid, and determined the amount of iron which passes into solution; the total amount of iron present was also found.

It appeared desirable to ascertain what proportion of this iron occurred in the ferrous state, and specimens of the fresh coal were treated with hydrochloric acid (10%) in an atmosphere of carbon dioxide. The excess of coal was removed by filtration in an atmosphere of carbon dioxide, and the amount of iron in the filtrate determined. It was found that the liquid contained a negligible quantity of iron in the ferric condition. The iron in the ferrous condition was determined by oxidation and subsequent titration with a standardised solution of titanous chloride. As this examination had to be performed on fresh samples of coal the results are not exactly parallel with those previously quoted. The preliminary results are, however, strictly comparative, and are given in the following table:—



TABLE VI.

	Total Iron. %	Iron (ferrous) extracted with hydro- chloric acid. %	Iron (ferrous) extracted with hydro- chloric acid. Percentage of total iron.
Rushy Park .. ..	0.494	0.176	35.6
Lower Mountain Mine ..	1.779	0.535	29.7
Arley .. ..	0.612	0.047	7.7
Bayliss .. ..	1.379	0.205	14.9

The results quoted in this paper are of interest as indicating that a distinct percentage of the iron in coal, which differs widely in different seams, is present in the ferrous condition in the ankerites, and in some other form of combination; it does not necessarily follow that the iron extracted by means of hydrochloric acid is present in the coal in the ferrous condition, since it may have been produced by the action of coal upon ferric compounds. In certain cases distinct oxidation of the ankerites can be detected within a month of the seam being obtained from the mine.

From a chemical standpoint it will be of interest to obtain information as to the relative rates of oxidation of ankerites of different compositions, and the effect of this oxidation upon the oxidation of the coal substance and of pyrites. It is not obvious which type will most readily oxidise, but upon the behaviour of these compounds will depend the degree to which coals will weather, and as a corollary their properties during storage.

Oxidation obviously produces a change in volume or thickness of the sheets of ankerite, and consequently is a factor contributing towards the disintegration of masses of coal. When coal has been allowed to stand for extended periods, oxidation of sheets of ankerites has been observed at a considerable depth into the coal. Up to the present no direct evidence has been obtained as to whether the primary heating of coal can be connected with the presence of ankerites, and the effect of manganese etc. upon the general action of the ankerites. At the same time, in view of the clearly recognised action of ferrous and manganese carbonates as catalysts it appears of considerable interest to examine the subject in the light of the information described in this paper. It would also appear that carbon dioxide may be an active agent in the oxidation of the compounds; we have found that ankerites are freely soluble in water containing carbon dioxide, and that the resulting solution rapidly undergoes oxidation with precipitation of the iron in the form of ferric compounds. In a mass of coal evolution of carbon dioxide is known to take place, and in the presence of any excess of moisture the conditions are such as to bring about the oxidation of the compounds, with carbon dioxide and water taking part in the reaction.

When coal containing ankerites is burned, bands of residue from the ankerite will remain separate from the inherent ash of the coal unless the temperature is sufficiently high to fuse the whole mass. The residual material from the ankerite will consist of highly infusible oxides. It would follow that coal which has been broken to a small size will contain the ankerite and silicious coal ash in more intimate contact than when larger sizes are used.

Analysts make a practice of quoting the colour of coal ashes, and it is well known that such ashes consist of a mixture of particles widely different in colour. We therefore made it a rule to pulverise coal ashes to a fine powder (1/200 mesh) in order to obtain an impression of the colour as a whole.

## DISCUSSION.

The CHAIRMAN suggested that the authors might determine how it was that the ankerites themselves were provocative of the oxidation of the coal material. If a portion of the coal were mixed with definite quantities of the ankerites it might be possible to determine how far the ankerites themselves were excitants of the coal oxidation. It might also be interesting to determine whether any of the particular seams of coal mentioned as containing manganese exhibited any greater tendency towards heating in the mass than those which were free from it, in view of the well-known catalytic effect which manganese possessed as an oxidation exciter.

Mr. H. L. TERRY said that as many of the analyses showed such low amounts of iron and manganese carbonates the term "ankerite" did not seem particularly appropriate. Certainly such terms as "dolomitic siderite" or "sideritic dolomite" were cumbersome and not to be recommended. He had often found coal partings to be of a dolomitic nature, but one sample he had had from the Lower Mountain Mine coal at Rishton, near Accrington, was pure calcite. Was this consistent with the authors' investigations? He noticed a great variation in the amounts of iron carbonate in the analyses quoted, and in many cases the manganese did not rise above what was ordinarily found in some limestones. Ankerites were not uncommon in the dolomitic beds of the lower carboniferous limestone in Derbyshire, and they were probably more widely distributed than was generally supposed. A peculiar manganese ore known as wad used to be obtained to a considerable extent at one time from pockets in the limestone adjacent to some of the mineral veins in Derbyshire. He had found that the oxides of manganese and iron were always mixed with magnesia, and he thought that the obscure origin of these deposits could be attributed to the action of carbonic acid on ankerite deposits. This was a matter he intended to probe further. At the Killeen copper mine, Co. Tipperary, the ore was associated with well crystallised ankerite showing about 30% each of the carbonates of calcium, magnesium, and iron, with 6-7% of manganese carbonate.

Mr. WILLIAM THOMSON enquired what was meant by the "faco" and the "end" of the coal, and also what process was adopted for estimating the carbon dioxide when the coal was treated with hydrochloric acid. Was it possible that some of the sulphides of iron in the coal would give off hydrogen sulphide which might be estimated as carbon dioxide? The authors had suggested various explanations as to why coal might become heated or take fire, but the usual explanation was that the oxidation of the pyrites produced the heat and fire.

Mr. STEVENSON asked whether the experiment had been made of grinding coal in a fairly high vacuum. Coal contained large quantities of occluded gaseous matter. Was there any possibility of carbon dioxide being occluded in the coal and set free on treatment with acid? If a sample of coal from one of the seams were treated with a ferric salt, say ferric chloride, and hydrochloric acid, a portion of the ferric chloride might possibly be reduced to the ferrous state. If so, that would lead somewhat to a modification of the view that the ferrous condition was the only one found.

Mr. McCULLOCH said that he had been unable to find any calcium or magnesium in the clays associated with some of the Lancashire coal seams. Could any theory be put forward as to how the ankerite came to be in the coal? If the ankerite was carried by a hard water, then it would appear that the surfaces of the under and over clays should

contain considerable quantities of calcium and magnesium.

Mr. BOOTH suggested that the ankerites were due to water infiltration from the limestone; the presence of comparatively high percentages of magnesium and manganese suggested this. The ferrous iron was no doubt derived from the chlorophyll present in the green parts of the coal measure flora. One would, of course, expect to find quite high percentages of ferrous carbonate. Coals which were liable to spontaneous ignition usually contained microscopically small yellow grains, as pointed out by Lomax. The possibility of these being chlorophyll grains should not be overlooked. The variation in the percentage of iron in the ankerites might be accounted for by the varying iron content of this coal seam, due to difference in constitution.

Mr. GROUNDS said that although Capt. Sinnatt has advanced no definite theory as to the connexion between ferrous carbonate, manganese carbonate, and spontaneous ignition, he had nevertheless hinted that there might be a connexion between these three factors. The South Wales bituminous coals contained 19–30% of volatile organic matter, as compared with 23–40% in the Lancashire coals. He thought it was generally agreed that spontaneous combustion was more liable to take place in coals containing a high percentage of volatile organic matter than in those containing a low percentage. Spontaneous ignition very rarely occurred with anthracites or steam coals. Ankerites were not present in the South Wales bituminous coals to anything like the same extent that they occurred in the Lancashire coals, and travelling westward from the Cardiff valleys towards the Swansea area, i.e., the anthracitic centre, there was a gradual transition in the same seam from a bituminous coal which contained 23% of volatile organic matter to an anthracite containing only 9%. At the same time there was another transition in the Eastern coals containing, say, 23% of volatile organic matter. There were quantities of ankerite visible, yet not so visible as in the Lancashire coals, while among the anthracites there were scarcely any ankerites at all. It seemed rather peculiar that the coals which possessed the highest volatile organic matter should have also the highest quantity of ankerites and also were the most liable to spontaneous ignition.

Mr. BLOCH asked whether Capt. Sinnatt had adopted any process to separate the alkali from the coal.

Mr. VARLEY referred to the figures in table III. showing that the result of exposing ankerites to air was to increase the ferric iron from 0.33% to 0.91%, whilst there was an increase in the amount of ferrous oxide from 9.29% to 10.58%; another remarkable point was the diminution of the silica from 0.91% to 0.17% after 12 weeks' exposure. Did the analyses shown give the composition of the same uniform sample before and after exposure to air, and if not could such results be relied upon for purposes of comparison?

Mr. TAYLOR mentioned that a piece of Mountain Mine coal weighing about 4 lb. was exposed to the air for about 13 months, at the end of which time the surface of the coal was covered with a white powder. When the coal was digested in water and the solution examined, the iron constituent was found to be in a ferrous condition. The solution also contained ammonium and sulphate in the proportions of one of ammonium to 40 of iron and 40 of sulphate. The oxidation or sulphating of coal might not be attributable to the iron in the coal.

Dr. A. C. DUNNINGHAM said that the authors' investigation into the nature of coal ash might be

of great service owing to the well-recognised fact that the practical value of coal depended not only on its theoretical heating value, but on the nature of its ash. It might also be of value in yielding information which would lead to a method of purifying coke. This was probably even now the cheapest form of carbon, which for some purposes was required in a very pure form.

Captain SINNATT, in reply to Mr. Varley, said that it was necessary to leave half of a piece of ankerite on the coal to stand for 12 weeks, whilst the other half was analysed. The same sheet of ankerite was used, but, as had been indicated in the paper, this material was not by any means constant in composition, and consequently the analytical figures for material one inch or so apart might be different. The silica in different samples might vary widely, as it was found deposited on the face of the material in local aggregations. It would be necessary, therefore, to accept these results until some better way of comparing the analyses could be adopted. The analyses only emphasised the point that considerable variations in the composition of the ankerites must be expected in a very short distance. The suggestion made by Mr. Allan was valuable, and if a method could be elaborated experiments would be carried out to test the influence of different percentages of manganese upon the oxidation of coal. In one part of the Ravine Mine the ankerite contained 4% of manganese, and although this observation was an isolated one, it was well known that the Ravine was the most liable to fire of any of the coals in the Lancashire coalfield. The remarks of Mr. Terry were of considerable interest, and it was most important that some name should be generally adopted for these white partings. The Lower Mountain Mine was, in certain parts, characterised by having a roof largely composed of marine fossils. It was quite possible, therefore, that intrusions of pure calcite would be found in it. The observations on the dolomites were especially pertinent. The terms "face" and "end" were used as applied in the coal industry, the face being the portion of the seam at which the collier worked, as the coal tended to fall in sheets towards him, owing to the line of weakness formed by the ankerites, etc. The end was at right angles to the face, and contained less of the partings. The method used for the determination of carbon dioxide was one elaborated by one of the authors (Sinnatt, Analyst, April, 1913); the contamination of the gas by hydrogen sulphide was not possible. The question of the influence of the pyrites upon the oxidation of coal was a debatable one, and had not been introduced into the paper, as Mr. Lomax of Bolton was at present engaged upon a systematic examination of the subject. Every endeavour had been made to eliminate occluded carbon dioxide. Each sample of coal was boiled with water for 20 minutes, in order to eliminate the occluded gases. The suggestion that the action of ferric chloride on coal should be examined was a valuable one; it was well known that iron pyrites had a reducing action on ferric chloride, but the exact action of the coal substance upon ferric chloride had received little attention. The authors agreed that the ankerites were distributed throughout the coal substance, and their thoughts tended to an opinion that some of the iron might be present in the coal in the form of an organic compound. The suggestion made by Mr. Booth that the iron was derived from the chlorophyll in the vegetation from which the coal was formed would account for practically all the iron found in coal seams. It was proposed, at a future date, to attempt to separate the ankerites by the froth flotation process. With reference to the remarks of Mr. Taylor, it would appear that he had been dealing with a deposit formed on the surface of the coal.

## NEW PROCESS FOR THE VULCANISATION OF RUBBER.

BY S. J. PEACHEY AND A. SKIPSEY.

The importance of the vulcanising process as one of the essential operations in the manufacture of all rubber goods is perhaps not fully appreciated, except by those who are directly connected with the industry; yet it may be said without exaggeration that but for the timely discovery of the process by Goodyear in 1839 the then newly-founded industry would not only have failed to attain its present position and magnitude, but would almost certainly have retrogressed.

Although it yields a vulcanised rubber of excellent quality, the Goodyear process itself possesses certain disadvantages, the chief of which are as follows:—(1) It necessitates the continuous use of steam both as a heating agent and as a medium for exerting pressure on the goods under treatment (and thus avoiding the development of porosity). (2) It is a comparatively slow process. (3) It restricts the manufacturer in his choice of filling and colouring agents. Organic filling agents, with very few exceptions, are destroyed or seriously deteriorated by the combined action of heat and sulphur, and in many cases by heat alone. Thus cheap and useful filling materials such as wood meal, leather, cotton, and wool wastes cannot be successfully employed in the manufacture of rubber goods. The manufacturer has to fall back on inorganic compounding materials such as barytes, chalk, china clay, kieselguhr and the like. Further, the majority of coal tar dyestuffs are destroyed or modified by the action of sulphur at a temperature of 140° C., so that the manufacturer has to confine himself to the use of mineral pigments such as zinc oxide, antimony sulphide, chrome green, and so on, a fact which explains the rather drab colours common to most rubber goods. Delicate tints are practically unobtainable.

The new process which forms the subject of this paper removes these restrictions and renders possible the production in the rubber trade of wholly new technical and artistic effects. The discovery of the process resulted from an investigation on the behaviour of rubber towards different forms of sulphur. Sulphur is remarkable for the number of allotropic forms which it is capable of assuming, in all three states of aggregation—solid, liquid, and gaseous—sulphur appears to be capable of varying its molecular complexity. An attempt was made to compare the action of these different forms of sulphur on rubber, and this attempt was interrupted by the discovery that the interaction of sulphur dioxide and hydrogen sulphide produces momentarily a form of sulphur which rapidly combines with rubber at the ordinary temperature, yielding an effective vulcanisation. The reaction between the two gases must take place in contact with the rubber or no vulcanisation results. In other words, the liberated sulphur is active only at the moment of its liberation, and it is fair to assume, therefore, that the effect is produced by atomic sulphur.

In applying the new process the rubber is exposed alternately to the action of sulphur dioxide and hydrogen sulphide. The gases readily diffuse into (probably dissolve in) the rubber, and there interacting produce active sulphur, which immediately combines with the rubber at the ordinary temperature, yielding a product wholly comparable with that obtained by the Goodyear hot process. Evidence is accumulating to show that the product actually superior in strength; this may be explained by the fact that the depolymerisation of rubber produced by heat is avoided in the new process.

The process appears to be of fundamental importance for the following reasons:—

(1) It is a true sulphur vulcanisation—as distinct from the sulphur chloride vulcanisation produced by Parkes' "cold cure."

(2) It eliminates the use of heat and to a great extent the use of mechanical pressure.

(3) It employs two gases, both of which can be produced on a large scale at a very cheap rate.

(4) It is rapid in action.

(5) It enables the manufacturer to employ organic filling agents which cannot be used in conjunction with the hot process or with the Parkes process (most organic materials are attacked and destroyed by contact with sulphur chloride).

A number of cheap and highly durable materials may be fabricated from various wastes in this manner and employed as floor and wall coverings, for boot and shoe manufacture, and for fancy leather goods and upholstery work. Further, in numerous manufacturing processes unconnected with the rubber industry the process renders possible the use of rubber as a binding agent for fibrous and granular materials as an alternative to the resins, bitumens, gums, and like substances which have hitherto been employed, with the result that the toughness and flexibility of the products are considerably increased.

(6) Coal-tar dyestuffs and even natural dyes like chlorophyll, which, with a few exceptions, are destroyed by the hot cure and also by the sulphur chloride cure, can be introduced into rubber mixings to be cured by the new process with the production of delicately-tinted materials hitherto quite unobtainable.

The process possesses the advantage of extreme simplicity, and its translation from the laboratory to the works should prove a simple matter.

The process can be extended to the vulcanisation of rubber in solution. If a solution of rubber in benzol or naphtha be saturated or partly saturated with hydrogen sulphide and mixed with a solution of sulphur dioxide in the same solvent, the liquid sets in a few moments to a stiff jelly, and on eliminating the solvent by evaporation a fully vulcanised rubber is obtained. The use of the mixed solutions for producing perfectly vulcanised seams and joints has proved highly successful, and inner tubes repaired by the new process have an excellent life.

Further, by the use of the solution process, reformed leather soles and heels may be attached to boots without the aid of stitching or nailing, and, indeed, a whole boot may be produced from the reformed leather without a single stitch being necessary.

During the past month or so certain modifications of the process have been devised, but these must form the subject of a later paper.

## DISCUSSION.

Mr. BAILEY asked whether the presence of the sulphur dioxide or the hydrogen sulphide in the rubber was likely to result in deterioration of the rubber in use. Also whether the removal of the gas was complete.

Mr. TOMKINSON asked if the water produced caused any porosity in the finished goods.

Mr. MARCROFT pointed out that one of the best red pigments was antimony sulphide, and that it usually contained a fair percentage of free sulphur. The same remark applied to ultramarine. Was that free sulphur affected during vulcanisation, or would it "sulphur up" afterwards?

Mr. LEON said that he had made experiments with the process described and had found that quite a lot of sulphuric acid was formed. How was this got rid of? He assumed that there was a certain amount of free sulphur in excess at the end of the process; did that "bloom" up and, if so, what method could be adopted for its prevention?

Dr. BLOCH asked how the new rubber compared with leather or with rubber vulcanised by the hot process in the wear of boots and shoes.

Mr. TERRY said that he had examined a sample of the rubber treated according to the authors' process, and after a lapse of six months in his possession he had found it to be perfectly sound. The problem in the rubber world was to find a really satisfactory "ageing" test. In order to obtain proof that rubber was properly vulcanised and not surface-cured the de-vulcanising processes could generally be employed. Rubber "cold-cured" by the Parkes process could always be de-vulcanised quite easily, especially in hot alkali, which removed the chlorine; but if the rubber cured by the "Peachey" process was treated by a hot alkaline solution there was practically no effect; the strongly vulcanised rubber remained intact. That was evidence of complete vulcanisation and not merely "surface curing," as in the Parkes process.

Prof. GREEN asked whether the process could be applied to the treatment of outer covers for tyres. It would appear that if it was possible to combine the strength of a fibrous material with the resiliency of rubber a great advance might be made in the wear of outer covers.

Mr. G. H. GRAY asked how it was proposed to manufacture a solid rubber tyre such as was used on a commercial vehicle.

Mr. PEACHEY, in reply, said that the two gases were introduced separately, both being comparatively easily soluble in the solid rubber, especially the sulphur dioxide. The amount of sulphur dioxide absorbed by rubber was surprising. Hydrogen sulphide was more than sufficiently soluble to yield a coefficient of vulcanisation up to, say, 5, which was higher than was required in practice. Adsorption was not relied upon at all, but absorption of the gas followed probably by solution. Generally speaking, excess gases, as far as could be judged by smell, were driven out of the rubber after about one hour's exposure. The practice had been followed throughout of giving the shorter sulphur dioxide treatment first and finishing up with the  $H_2S$  in excess, so that there was very little possibility of free  $SO_2$  remaining and practically no danger of free acid forming. To get a fully vulcanised rubber it was only necessary to introduce some 2½% of sulphur; therefore the amounts of the two gases required to vulcanise a mixture containing, say, 50% of rubber were surprisingly small, and there was only a negligible amount of water produced. The water diffused out of the rubber quite rapidly; it never existed in the liquid form in the finished product, and the vulcanisation need not be followed by any drying operation; mere exposure to the air for a few hours, at any rate, caused all necessary

elimination of water. As regards the free sulphur present in antimony sulphide and ultramarine, it was disadvantageous to have free sulphur present in a mixing which had to be vulcanised by the new process. He supposed it tended in some way sympathetically to convert the atomic sulphur into molecular sulphur. In all mixings which were made for the new process ordinary sulphur would not be present, and the use of antimony sulphide would be especially avoided, as that substance could be replaced by much better and brighter colours. In the "dry" treatment the rubber or rubber mixing to be vulcanised was exposed to sulphur dioxide for 10 minutes, after which a very short exposure to the air was given to remove the adsorbed gas from the surface. The material was then introduced into another chamber where it was exposed to the hydrogen sulphide for 20 to 30 mins. In the case of "solution vulcanisation," it was very easy to prepare standard solutions, and in practice a standard solution of sulphur dioxide was prepared by weight. It was convenient to use a solution containing 0.8% of this gas in benzene, and to saturate a 10% rubber solution with hydrogen sulphide, and mix four volumes of the hydrogen sulphide solution containing the rubber with one volume of the benzene solution. The actual proportion of the two gases interacting was theoretical. Small amounts of free sulphur were invariably formed in the rubber. The combination was not quite complete, as apparently a small amount of the atomic sulphur was changed into molecular sulphur, but the amount was small compared with the amount that was left in the rubber by the "hot" process. It was a matter of surprise to learn that Mr. Leon had found acid in his samples; possibly he had used the sulphur dioxide in excess, or at any rate not in sufficient deficiency. In his own experiments the hydrogen sulphide had been invariably kept in excess, and by treating the rubber first with sulphur dioxide and then with hydrogen sulphide the formation of any trace of free acid could be avoided. If, however, faulty working led to the formation of a trace of free acid the material could be treated with ammonia, just as in the sulphur chloride process. The question of the treatment of rubber of one inch thickness was rather beyond the present limits of the process. The porosity of a mixing was actually greater when it was fairly heavily loaded, and it was surprising what penetration was obtained. Although exact diffusion figures were not available, it might be assumed that both sulphur dioxide and hydrogen sulphide diffused into rubber at least as rapidly as carbon dioxide, and in the case of sulphur dioxide more rapidly. They would not attempt by the new process to deal with material one inch thick, but would avail themselves of the new method of building up which had become possible as the result of the new solution process. It was possible now to build up material of any thickness after it had been vulcanised in sheets and to get a solid mass in which the joints were of a strength equal to that of the material itself. The treatment of thick articles would, therefore, involve new methods of building up, but it was quite desirable that such new methods should be introduced. The leather compounds prepared by this process were 2½ times as durable as new leather.

## Liverpool Section.

Meeting held on November 26, 1920.

## THE CORROSION OF COKE OVEN WALLS.

BY A. E. FINDLEY.

The work described in this paper was undertaken with the idea of throwing some light on the effect of certain ingredients in the coking slack on the corrosion of the oven walls. The ingredients which were suspected of producing the most harmful effect were salt, iron, and moisture.

For the purposes of this work the total amount of salt present in the coals examined was not determined, but only that amount which could be removed by washing 200 grams of each coal by percolation with cold water under exactly similar conditions. The main object of this washing was to test the retentive capacity for water of different coals (as sent to the ovens), and also to show to what extent this retentive capacity depended on the fineness of the coals after crushing. Subsequently each coal was washed until 100 c.c. of the wash water required 2 c.c. or less of  $N/50$  silver nitrate solution. In each case the same amount of wash water was used on a definite weight of coal. The quantity of salt extracted was proportional to the quantity actually present in the coal within certain limits.

the finest crushed slack was used, the coal in the hoppers is not saturated with water. The sub-joined table indicates the first stages of the work for four different Yorkshire coking slacks.

The relative retentive capacities for water in the table are purely empirical and are based on the rate of draining under similar conditions.

The following conclusions may be drawn from the table:—

(1) As the amount of salt increases the durability of the oven walls decreases.

(2) If the amounts of both salt and iron present are high, as at Waleswood, durability seems to be still further diminished.

(3) Iron and moisture may be present in relatively large amounts, but this does not appreciably affect the durability of the oven walls if the amount of salt is very low, as at Hoyland Silkstone.

(4) The higher temperature in vertical flues of a regenerative oven system tends to shorten the life of the oven walls.

W. J. Rees has shown (*Cf. Ceram. Soc., Spring, 1920*) that iron may be carried right through and out of a fireclay brick by means of salt at  $950^{\circ}\text{C}$ . Working side by side with Rees I have endeavoured to throw some light on the mode of transference of iron from coal into the fireclay of the oven walls, because Rees has shown (*Trans. Ceram. Soc., 1918*) that spalling mainly takes place at a layer of hæmatite formed some distance inside the bricks; he explains the presence of hæmatite by assuming that the iron is carried into the bricks as chloride until it meets with oxidising conditions.

## Tests on four coals in South Yorkshire.

Colliery.	Type of ovens.	Salt. %	$\text{Fe}_2\text{O}_3$ . %	Water. %	Ash. %	Relative retentive capacity for water.	Relative fineness.	Life of ovens.
Waleswood ..	Simon-Carves, (a) regenerative (b) waste heat.	0.142	1.45	15.5	8.75	8	4.1% above $\frac{1}{16}$ " 20.5% above $\frac{1}{16}$ " less than $\frac{1}{8}$ " 21.1% above $\frac{1}{16}$ " less than $\frac{1}{8}$ " 54.3% less than $\frac{1}{16}$ "	(a) 18 months (b) 24 years roughly
Monkton Main..	Simon-Carves, waste heat.	0.086	0.82	11.5	9.0	3.4	16.25% above $\frac{1}{16}$ " 41.5% above $\frac{1}{16}$ " less than $\frac{1}{8}$ " 19.75% above $\frac{1}{16}$ " less than $\frac{1}{8}$ " 25.5% less than $\frac{1}{16}$ "	Roughly 3 years.
Wharfedale Wood- moor	Simon-Carves, regenerative.	0.038	0.71	8.4	4.9	1	23.8% above $\frac{1}{16}$ " 20.6% above $\frac{1}{16}$ " less than $\frac{1}{8}$ " 14.3% above $\frac{1}{16}$ " less than $\frac{1}{8}$ " 27.3% less than $\frac{1}{16}$ "	Roughly 4 years.
Hoyland Silkstone	Semet-Solvay, waste heat.	0.023	1.09	12.4	8.05	29	1.43% above $\frac{1}{16}$ " 9.34% above $\frac{1}{16}$ " less than $\frac{1}{8}$ " 12.05% above $\frac{1}{16}$ " less than $\frac{1}{8}$ " 65.98% less than $\frac{1}{16}$ "	No repairs since 1917 when ovens were built.

J. W. Cobb has shown that, however finely a coal be crushed, washing with cold or boiling water does not remove all the salt, and that further prolonged boiling with water and with dilute nitric acid will yield further quantities of salt; consequently, the figures given for salt in this work are better for the purposes in view than figures taken from the average results over one year's working at the coke ovens using the coals examined, because they have been obtained under almost exactly similar conditions, whilst the conditions for the determination of salt at different coke ovens are very variable.

The amount of water in the coal going to the hoppers will be dependent initially on the size of the unwashed coking slack; the amount of water retained in the coal after crushing (i.e., the amount of water which does not drain out, however long the coal remains in the hoppers) will depend entirely on the fineness of the crushing. The results obtained have proved this conclusively, and also that at the Hoyland Silkstone Colliery, where

The following experiments were carried out by heating 5 g. of thoroughly mixed coal (ground to pass a 30-mesh sieve) in silica tubes to  $900^{\circ}\text{C}$ .— $950^{\circ}\text{C}$ . under the conditions stated:—

Substance heated.	Period in hours.	Atmosphere.	$\text{Fe}_2\text{O}_3$ found in ash, calc. on original coal %
Coal A	4½	Air	1.56
Coal + $\text{CaCl}_2$	4½	Steam and air	(a) 1.46 (b) 1.42
	4½	Air	1.42
Coal	4½	Steam & air	1.43
*Coke	4½	Steam & air & $\text{HCl}$	1.28
	4½	Coal gas	1.53
Coal B	4½	Air	1.44
Coal, $\text{MgCl}_2$ , & $\text{NaCl}$	4½	Air	1.32
*Coke, $\text{CaCl}_2$ , & $\text{NaCl}$	4½	Coal gas.	1.32
Coal, $\text{CaCl}_2$ , & $\text{NaCl}$	4½	Air & steam	1.42
*Coke	45	Coal gas	1.44

\* Where coke is quoted it is the coke from 5 grams of coal.

It will be seen that the maximum loss of iron is obtained by burning the coal in an atmosphere of hydrochloric acid vapour and air. Chlorides appear to be capable of removing iron in the presence of air and steam or of coal gas, but coal gas alone is not effective in removing iron from coal or coke. Of the above results those with chlorides cannot be regarded as absolutely conclusive, as I have found that the majority, although all pointing to the fact that iron is removed, are not outside the limits of experimental error; for definite proof of the removal of iron it is necessary to depend on the result obtained with hydrochloric acid which, in my opinion, is quite beyond the limits of probable error of experiment. There is no doubt that chlorides have the same effect in the presence of gas and steam at the temperature of the ovens, as most chlorides likely to be present in coal will yield hydrochloric acid under the conditions existing in coke ovens.

Applied Chemistry Laboratory,  
The University, Sheffield.

## Communications.

### A NEW METHOD FOR THE PRODUCTION OF CELLULOSE ACETATE.

BY W. LEIGH BARNETT, B.Sc., A.I.C.

A variety of substances have been employed as catalysts of the acetylation of cellulose, the chief of which are strong dehydrating agents, such as sulphuric acid, phosphoric acid, zinc chloride, chlorides and oxy-chlorides of sulphur and phosphorus, dimethyl sulphate, chloroacetic acids, etc. Chlorine does not act readily unless under special conditions, and then only very slowly. Combined in the form of chloroacetic acids, large quantities are required compared with the weight of cellulose employed. Many of these catalysts possess the disadvantage that they give coloured or cloudy solutions owing to decomposition and degradation of the cellulose ester. Sulphuric acid, which is largely employed, tends to give dark-coloured solutions and also to hydrolyse the product, in addition to which the properties of the resulting esters vary considerably. Further, the films are generally brittle owing to the fact that all the sulphuric acid cannot be removed successfully. It is claimed that addition of substances which neutralise the effect of the acid, or other condensing agent, keep the syrupy solutions of cellulose acetate in good condition and prevent them from giving brittle products. A distinct improvement in the acetylation of cellulose results from the use of sulphuryl chloride as catalyst (Chem. Fabrik von Heyden, F.P. 24,382 of 1910), whereby superior cellulose acetate solutions result, and these solutions are not decomposed by the sulphuryl chloride.

For an investigation into the esters of cellulose it was necessary to make use of cellulose acetates which had been prepared in such a way as to ensure the minimum amount of change in the parent complex. All of the ordinary methods for acetylating cellulose were rejected on the grounds that either the ester solutions were coloured after acetylation had occurred, or that the solutions were not brilliantly clear, and for these reasons there was no guarantee that the esters when isolated would not contain products of degradation. By using a mixed catalyst of chlorine and sulphur dioxide I have found that satisfactory results are obtained with the minimum amount of change in the parent complex. If cellulose be treated with a suitable mixture of glacial acetic acid, acetic anhydride, and a trace

of chlorine, practically no acetylation results unless the mixture be heated for some time. Similarly, if sulphur dioxide be substituted for the chlorine, no acetylation occurs, and if the mixture is heated it takes place to a smaller extent than in the case of chlorine. If, however, traces of both chlorine and sulphur dioxide be introduced, either separately or together, into the mixture, acetylation proceeds to a remarkable extent, and a perfectly clear, colourless solution, or even jelly results. By suitably choosing the conditions for the reaction it is possible to obtain a great variety of pure esters, possessing valuable properties of strength, film flexibility, transparency, and solubility. Acetylation proceeds at any temperature, and as no signs of discoloration occur, owing to the great ease of control of the reaction, no special precautions are necessary. The combined use of chlorine and sulphur dioxide is equally efficient in producing other esters, such as the butyrate, benzoate (using benzoyl chloride in acetic acid), etc.

The great reactivity of this mixed catalyst is probably due to the fact that first the cellulose itself acts as a condensing agent for the chlorine and sulphur dioxide, which produce small amounts of sulphuryl chloride in intimate contact with the cellulose, and these traces of sulphuryl chloride, being produced *in statu nascendi* under such conditions, are much more effective in promoting the subsequent acetylation.

A large number of experiments were carried out in order to ascertain the best conditions for the acetylation of various kinds of cellulose. Swedish filter paper, bleached and grey American cotton yarn, and cotton wool were employed, whilst variations in the method of adding the catalysts together with alterations in relative quantities and conditions of temperature were introduced. The best method for isolating the cellulose ester from the acetic acid solutions was found to consist in the addition of chloroform or acetone to the reaction solution, followed by mixing with an excess of water. When the volatile solvent is distilled out the cellulose acetate is gradually precipitated, due to the rising of globules of the chloroform solution through the water. The globules become covered with a fine film of precipitated cellulose acetate, and on stirring these are disintegrated. By this means the ester is obtained in the form of a fine white powder, and can easily be washed free from acid. The products can be dried at 100° C. for several days, and do not show any signs of decomposition or darkening in colour. The properties of film strength etc. are not impaired by continued heating.

The product depends on the conditions of acetylation. If the temperature be kept below 65° C., and only a trace of sulphur dioxide be employed, the product is chiefly a cellulose diacetate. If the ratio of chlorine to sulphur dioxide be nearer to unity then the product is mainly cellulose triacetate, particularly if the temperature be allowed to rise above 65° C.

The following table shows the nature of the products obtained after varying intervals:—

Time. Hrs.	Yield. %	Acetic acid. %	Cellulose. %
1 .. ..	178	63.9	58.9
2 .. ..	173	63.7	58.1
24 .. ..	174	—	—
48 .. ..	173	63.3	51.5
72 .. ..	173	65.0	48.9
72 .. ..	176*	61.9	48.8

\* In this case there was formed in addition a small quantity (1.2%) of an acetate insoluble in acetic acid. Theoretical yield for cellulose triacetate, 178%; C 49.9%, H 5.6%. The product contained C 49.2%, H 5.3%.

In one experiment 200 g. of acetic acid, containing only sufficient chlorine to colour it, was poured on to 50 g. of filter paper, and after standing for some time 250 g. of acetic anhydride was added, followed by the passage of a few bubbles of sulphur di-



oxide. The temperature rose spontaneously and was kept down to a maximum of 65° C. by cooling with water. Within an hour a clear solution resulted. After standing over night it set to a firm jelly, which was diluted with acetic acid and separated by means of chloroform. The fine white powder ultimately obtained was soluble in acetone, chloroform, pyridine, hot nitrobenzene, aniline, and slightly soluble in a mixture of alcohol and benzene. Analysis showed it to be a diacetate (C, 48.17; 48.93%; H, 5.66, 5.73%; theory requires C, 48.78%; H, 5.73%). Acetic acid by hydrolysis of an acetone solution (*cf. infra*, the analysis of cellulose acetates), 48.81–48.98%; theory requires 48.8%.

In another experiment 5 g. of filter paper was immersed in 20 c.c. of glacial acetic acid, and 20 c.c. of acetic anhydride containing 0.32 g. of chlorine added, followed by 2 c.c. of acetic anhydride containing 0.26 g. of sulphur dioxide. The mixture rapidly gelatinised, and in less than five minutes solution was complete. After a further five minutes' stirring 20 c.c. of chloroform was added and the ester separated as usual. The product formed fine white flakes of cellulose triacetate (62.4% of acetic acid; theory requires 62.5%). Using the same quantity of cellulose, 20 c.c. of acetic acid, 45 c.c. of acetic anhydride containing 0.066 g. of chlorine and 1 c.c. of anhydride containing 0.069 g. of sulphur dioxide, and warming the mixture slightly, a fine pulp was produced after 10 minutes, which rapidly gelatinised and gave a clear viscous solution from which the ester (acetic acid 84.2%) was isolated.

Preliminary experiments showed that acetylation proceeded best when the weights of chlorine and sulphur dioxide were approximately in the ratio of their molecular weights. This was ascribed to the formation of sulphuryl chloride, and it was therefore considered that by working at its boiling point (69° C.) the reaction would be greatly expedited, since then the sulphuryl chloride would tend to remain gaseous and the escaping bubbles should aid in the rapid clarification of the solution. A mixture of 2 g. of filter paper with 10 c.c. of acetic acid was heated to 69° C., 6 c.c. of acetic anhydride containing 0.23 g. of chlorine added, and then 3.7 c.c. of the anhydride containing 0.25 g. of sulphur dioxide, the temperature being maintained at 69°–70° C. by means of a thermostat.

It is interesting to note that both the acetic acid figures and the values for the recovered cellulose fall off with increase of time of acetylation. The decreasing figures for the cellulose recovered may be taken to indicate some change of the cellulose complex, and although the yields of ester isolated at different stages are the same, this can be explained by the fact that esters from modified cellulose are insoluble in water. The cellulose recovered in the above cases was found to possess the characteristic properties of cellulose, being insoluble in any of the solvents for cellulose acetate and soluble in ammonium from which it was reprecipitated by acids.

Cotton wool and bleached and grey American cotton yarn gave similar results to those described for filter paper.

Parallel experiments were made using cotton wool and filter paper. The air-dried samples were immersed in cold diluted Fehling's solution for 30 mins. and washed free from copper solution, and the absorbed copper removed by digestion with dilute acetic acid. The cotton (7.33% moisture) was found to have absorbed 0.83% of copper and the filter paper (5.73% moisture) 0.54% on an air-dry basis (these amounts of copper are proportional to the amounts of cellulose hydrate present. The residual cellulose was boiled with dilute Fehling's solution for 15 mins., the absorbed copper removed, and the products acetylated at 69°–70°

C.; the following results were obtained (on an air-dry basis):—

	Cotton.	Filter paper.
Copper, %	1.50	8.82
Residual cellulose, %	92.1	87.4
Yield of ester (total), %	170	163

The filter paper cellulose acetylated more vigorously, but the cotton gave a solution of better colour and higher viscosity than the filter paper. After standing for 24 hours a small amount of the cellulose acetate insoluble in acetic acid was precipitated, amounting to 10.6% in the case of the cotton product and 3.9% in the case of the filter paper. These results indicate that the presence of much cellulose hydrate lowers the yield of cellulose triacetate, due no doubt to the formation of soluble products of degradation of the cellulose.

The acetylation of cellulose in presence of a large quantity of water was examined with bleached and with grey American cotton yarns. The absorbed copper due to cellulose hydrate was determined by the action of Fehling's solution in the cold. The wet cellulose was pressed after treatment with the acetic acid and washing with water, and the damp cellulose then acetylated at 65° C. for an hour. Acetylation was more noticeable in the case of the grey cotton. The products were separated by means of chloroform, and the dry products, which retained the fibrous condition, were re-acetylated, giving clear viscous solutions, that from the bleached cotton being superior. The bleached cotton gave 18% of an ester insoluble in chloroform. The following figures show the experimental data:—

		Bleached.	Grey.
Moisture, %	.. ..	0.65	7.20
Copper % on dry cellulose	.. ..	0.83	0.26
First acetylation, yield	.. ..	103	131
Second acetylation, yield	.. ..	150	142
Chloroform-soluble portion, %	.. ..	48.68	47.00
Acetic acid, %	H% .. ..	5.73	5.03
Recovered cellulose, %	.. ..	49.5	51.2

These results appear to indicate that the main ester from the bleached cotton is a diacetate, whereas that from the grey is a mixture of esters approximating to the diacetate.

#### Note on the analysis of cellulose acetate.

Several methods of hydrolysis of the ester and estimation of the acetic acid by subsequent distillation and titration of the volatile acid, have been suggested. These all possess the disadvantage that the cellulose regenerated by the reaction itself decomposes at the same time, giving rise to volatile acids for which a correction is necessary. It was found that, in the case of acetone-soluble cellulose acetates, the estimation can be greatly simplified. Briefly the method consists in dissolving the weighed cellulose acetate in acetone, hydrolysing the ester in the cold by shaking with a known amount of standard caustic soda in a stoppered flask, allowing to stand for a day, diluting with water, and titrating the excess alkali with standard acid and phenolphthalein. A blank experiment is carried out, using a similar quantity of acetone to which a known weight of cellulose (Swedish filter paper) has been added, and also a blank on a similar volume of acetone. From the two blank estimations a correction for any acidity in the acetone, and also for any decomposition of the cellulose, is made.

The following results with cellulose diacetate give the necessary data:—

Material.	Weight.	Acetone, c.c.	N/10 NaOH, c.c.	N/10 H <sub>2</sub> SO <sub>4</sub> , c.c.	Acetic acid, %.
Ester	0.3167	30	47.0	18.85	48.98
Cellulose	0.3387	30	47.0	17.05	48.81
Blank	0.3000	30	47.0	44.20	—
Blank	—	30	47.0	45.90	—

The loss due to the 0.3 g. of cellulose is thus 1.7 c.c. N/10 NaOH. Inspection of the amount of

caustic soda neutralised by the ester indicates that the ester is approximately a diacetate, and therefore the regenerated cellulose initially amounted to approximately two-thirds of the weight of ester taken. Hence the number of c.c. of  $N/10$  NaOH equivalent to that neutralised by the regenerated cellulose from the first estimation is

$$\frac{1}{3} \times (0.3167/0.30) \times 1.7 = 1.2 \text{ c.c.}$$

This gives the alkali that is equivalent to the acetic acid as being 45.9–18.85–1.2 c.c.  $N/10$  NaOH = 48.98% acetic acid. Similarly the correction for the cellulose from the second case is found to be 1.3 c.c. This being allowed for gives 48.81% of acetic acid. When the same ester was submitted to alkaline hydrolysis, and the acetic acid determined by distillation, the results were from 43.54 to 44.07%.

In the case of samples of the triacetate the figures will show that the approximate amount of acetic acid is 60%; hence the regenerated cellulose will be about 55%. The correction is therefore based on this and the results worked out as above.

The estimation becomes simpler if the same weight of ester be taken for all estimations, as then the correction to be applied will be uniform.

The ordinary methods of estimation of the acetic acid, which involve distillation and titration of the acid in the distillate, are not accurate enough to detect differences of the order of 1% of acetic acid which are readily indicated by this more exact method.\*

My thanks are due to the Department of Scientific and Industrial Research for permission to publish these results, also to Professor Sir W. J. Pope for allowing the investigation to be conducted in the Chemical Laboratory, Cambridge.

## POSSIBLE DEVELOPMENTS IN HYDROGEN MANUFACTURE.

BY ERIC K. RIDEAL, D.SC., M.A.

The processes now in operation for the manufacture of hydrogen from coal or coke as raw material have centred round the steam-iron process for small scale and the water-gas catalytic process for large manufacturing units. The electrolytic production of hydrogen using coke as the source of power is only economically possible when the hydrogen is formed as a by-product in some electrochemical industry or in special cases where its actual generation and consumption may be confined to interpeak periods on the electrical load.

The Linde-Frank-Caro process of separating hydrogen from water-gas by liquefaction possesses the advantage of delivering hydrogen free from all impurities except carbon monoxide, and conveniently under pressure. The two chief disadvantages of the system are, firstly, the cost of operation and upkeep of the relatively complicated, high-speed machinery involved in the liquefaction plant, which can be partly set off by the proceeds resulting from the installation of an oxygen recovery plant on the liquefier; and, secondly, the high carbon monoxide content (2–4%) of the hydrogen when operating under conditions which will furnish the maximum yield of hydrogen (i.e., 80–85% of the total available in the water-gas). This gas can of course be utilised for many industrial purposes, e.g., for filling balloons or hardening fats, but not in those catalytic processes in which low-tempera-

ture hydrogenation is desired, such as in the preparation of the finer hydrogenated oils and hexahydrobenzene, or where the carbon monoxide exhibits a marked inhibiting action, as in the synthesis of ammonia using an iron-hase catalyst, or again where it acts specifically as a poison as in the synthesis of ammonia with an irreversible catalyst such as sodamide or uranium carbide. Using a water-gas of average composition (42% CO, 49% H<sub>2</sub>, 4% CO<sub>2</sub>, 5% N<sub>2</sub>, and CH<sub>4</sub>) 2.4 volumes would be required to produce one volume of hydrogen. The general utility of the method can only be extended by some effective method for the removal of the carbon monoxide, a point which will be referred to later.

In the steam-iron processes the two representative types are exemplified in the Lane or retort, and the Messerschmidt or block type generators. Under normal conditions these operate between temperatures of 650° and 850° C., and it is interesting to note how far existing practice falls short of the theoretical possibilities of the method. The utilisation of a contact material more reactive than spathose has been suggested in many patents. Such material must be superior to spathose in two respects; firstly, it must operate at a lower temperature, or more rapidly at the same temperature; and, secondly, it must present a larger active surface under operating conditions.

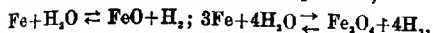
To produce some 2000 ch. ft. per hour of hydrogen 7000 kg. of spathose is required; if complete conversion ( $\text{Fe} \rightarrow \text{Fe}_3\text{O}_4$ ) were effected only 75 kg. would be necessary. The actual yield indicates an output of only 14 ch. ft. of hydrogen per ch. ft. of retort space per make, and a 1% conversion of the spathose. Taking average spathose of 2 in. diameter and mean density 5, the calculated effective conversion depth is of the order of 0.1 mm.; microscopical examination reveals a penetration of 2–3 mm., indicating that but little of the surface is reactive and that the rate of chemical action is consequently partly limited by diffusion of the reactants into the interior. Since a 50% loss on screening after two months operation is by no means uncommon, it is evident that there is considerable room for improvement in this respect. The replacement of the spathose by various forms of activated irons, especially iron-manganese and iron-copper, certainly results in increased yields for short periods, but the enhanced activity at the high temperatures of operation is usually short-lived, owing chiefly to irreversible poisoning during the reducing phase, and loss in catalytic activity due to the thermal treatment which the material undergoes especially in its exothermic phase. At the period of the armistice experiments were being commenced by the Munitions Inventions Board and Department of Explosives Supply to ascertain how far this small surface yield could be increased by an inversion of the process; i.e., instead of passing water-gas and steam over iron oxide, it was attempted to blow finely divided iron oxide through a pipe by means of hot water-gas and transfer the reduced oxide to another pipe, also maintained at suitable temperatures, for oxidation with steam. On paper this scheme presents striking possibilities not only as regards economical utilisation of the spathose—a relatively secondary matter—but also in respect of the size of plant required and the almost certain increase in the activity of the material in this form, permitting lower operation temperatures and an economy in both water-gas and steam. Several types of mechanical devices for the separation of dusts have recently been designed which should provide those interested in the development of such a process with sufficient working data for a suitable design of plant.

The economy of steam and water-gas in the steam-iron process can likewise be examined by comparison

\* Recent determinations show that even better results are obtained by using 25 c.c. of acetone,  $N/1$  alkali, and  $N/10$  acid. The correction in this case for the regenerated cellulose is smaller, and for comparative purposes can be neglected.

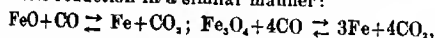
with the theoretical consumption on a basis of unit hydrogen production.

The reactions indicated by the equations,

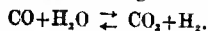


have been examined in detail by numerous investigators, notably by Deville (Comptes rend., 1870, 76, 1105, 1201), Dehray (*ibid.*, 1879, 88, 1341), Preuner (Z. physik. Chem., 1904, 47, 385), and Chaudron (Comptes rend., 1914, 159, 237). The various data do not show complete agreement as to the heats of reaction calculated by means of the van't Hoff isochore, but both reactions are strongly exothermic (from 32.1 to 44.1 kg.-cals. per g.-mol.). The heat liberated during the steaming phase can generally be noted on the pyrometer and is entirely lost to the system. The effluent gas will of course consist of steam and hydrogen, and during the complete make the ratio of steam to hydrogen can never fall below the equilibrium values as determined by the above reversible equations; these are 0.176:1 at 440° and 1.0:1.0 at 1500° C. In actual practice, especially towards the end of the steaming phase, a great excess of steam will be present in the effluent gas. Some actual observations on a Lane setting indicated a steam:hydrogen ratio of 1.66:1.0 at the commencement and 6.5:1.0 at the end of a good make, the average being about 3.0:1.0. Thus during the major portion of the make nearly ten times the amount of steam necessary for the reaction was passing through the system, entailing loss both in material and in sensible heat. This loss could doubtless be minimised by fixing a suitable automatic valve on the steam inflow.

The reactions occurring during the reducing phase are much more complicated than those in the steaming phase. Apart from numerous secondary reactions common to both phases, the main reactions on reduction are unknown. It is, however, practically certain that the hydrogen reduces the oxide in accordance with the above equations, but it is as yet uncertain whether the carbon monoxide effects reduction in a similar manner:



or whether it operates in a secondary manner due to the intermediate formation of hydrogen according to the well-known water-gas reaction,



Although carbon monoxide is a more powerful reducing agent than hydrogen below 850° C., when the equilibrium value constant of the water-gas reaction becomes unity (Henry, Phil. Mag., 1836, 39, 324; Fay and Seeker, J. Amer. Chem. Soc., 1903, 25, 641), yet in the absence of moisture it is extremely sluggish. Moisture acts as a catalyst, possibly because the hydrogen formed is a more rapid but less powerful reducing agent than carbon monoxide. An analysis of the spent water-gas from a Lane setting indicated, however, that the water-gas equilibrium was obtained at the operating temperature of 650° C. It is thus possible with the aid of the data on the system hydrogen, water, iron and its oxides, together with those on the water-gas reaction to calculate the theoretical consumption of water-gas during the reducing phase. It may be noted in passing that these data are at variance with those of Baur and Glaessner (Z. physik. Chem., 1903, 43, 358), Schenck (Ber., 1905, 38, 2132; 1907, 40, 1704), and Falcke (Z. Elektrochem., 1906, 22, 300) on the reactions in blast-furnaces. The cause of the differences is possibly due to the deposition of carbon or the formation of carbides in the blast furnace. The reduction of iron oxide by carbon monoxide appears to be an exothermic reaction at these relatively low temperatures.

The theoretical consumption of water-gas per volume of hydrogen produced has been calculated by Dr. Taylor and the writer, with the following results:—

Cycle $\text{Fe} \rightleftharpoons \text{Fe}_3\text{O}_4$			
Temperature, °C.	% H <sub>2</sub>	% CO	Vols. water-gas per vol. hydrogen.
650	30	41	2.55
750	33	40	2.77
850	41	35	2.83

If, on the other hand, the ore does not operate through the cycle  $\text{Fe} \rightleftharpoons \text{Fe}_3\text{O}_4$ , but through the cycle  $\text{FeO} \rightleftharpoons \text{Fe}_2\text{O}_3$ , the consumption of water-gas will be lower, although a smaller yield per kg. of ore will naturally result.

Cycle $\text{FeO} \rightleftharpoons \text{Fe}_2\text{O}_3$			
Temperature, °C.	% H <sub>2</sub>	% CO	Vols. water-gas per vol. hydrogen.
650	42	64	1.8
750	56	70	1.6
850	60	80	1.4

The actual consumption varies between two and three volumes.

The utilisation of the carbon monoxide fraction of the water-gas for reduction purposes has been suggested by several investigators, notably by Jaubert. As has already been indicated, the reaction appears to be exothermic at 650° C., but on the other hand the reaction velocity may be exceedingly low. In addition, precautions have to be taken against the precipitation of carbon on the iron through the secondary reaction  $2\text{CO} \rightleftharpoons \text{C} + \text{CO}_2$ . This latter difficulty can, of course, be readily remedied by application of one of the three general methods which have been suggested. All of these rely upon the maintenance of a suitable ratio of carbon dioxide to carbon monoxide in the reducing gas so as to prevent the precipitation of carbon. According to the data of Boudouard (Ann. Chim. et Phys., 1901, VII, 24, 5), and Rhead and Wheeler (J. Chem. Soc., 1910, 2179, and 1911, 1141), the ratio carbon dioxide/carbon monoxide is 1.67 at 650° C., 0.72 at 700°, 0.60 at 750°, and 0.07 at 800°. These conditions can easily be produced by operating at very high temperatures, where it will be noted that the ratio sinks to such a small value that the carbon dioxide present in the water-gas or the small quantity produced as it enters the retort is sufficient for the purpose. In general, however, this is not an economical method owing to the short life of the retorts and to the increase in the radiation loss from the bench. The higher CO<sub>2</sub>/CO ratio necessary at the lower temperatures can be obtained either by the admission of a little air, as in the block type generator, where partial internal heating obtains, or by the admission of steam during the reducing phase, when an increase in the carbon dioxide content results through the operation of the water-gas reaction. Alternatively the same result may be obtained by passing the mixture of steam and water-gas through a pre-converter containing catalytic material maintained at a suitable temperature. If more careful investigation supports Jaubert's views as to the merits of carbon monoxide as a reducing agent it would be a comparatively simple matter, provided that due attention be paid to the CO<sub>2</sub>/CO ratio, to produce such a gas by the passage of spent water-gas, after removal of the steam by condensation, back through the water-gas generator. Such a gas is clearly much more economically produced than "straight" blue water-gas.

It is evident that with iron oxide as contact material no great improvement over existing practice in water-gas consumption is to be expected, although a marked improvement in steam consumption is theoretically possible. Economy of steam can readily be effected by the employment of activated irons, as is exemplified by the following figures obtained with spiegeleisen (18% Mn) as contact material.

Temp., °C.	Vols. steam per vol. hydrogen in effluent gas.
600	2.0
650	1.8
660	1.6
720	1.25

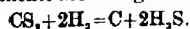
To reduce the consumption of water-gas involves the utilisation of an oxide more easily reducible than iron; other considerations, such as cost and fusibility of the reduced metal, have apparently militated against the adoption of such materials.

The fuel employed for keeping the retort system hot must also be included on the debit side in hydrogen manufacture. From this point of view the block type of generator is far superior to the retort type, but the hydrogen produced by the former is usually not so pure owing to the difficulties associated with the scavenge. Experiments in Sweden on a typical retort setting of brick and magnesite showed a radiation loss of some 470,000 kg.-cals. per hour. With an average water-gas of 75 kg.-cals. net per cb. ft. a consumption of 6300 cb. ft. per hour would be necessary to make up the deficit. During this period 4000 cb. ft. of hydrogen is produced with an internal consumption of 10,500 cb. ft. of water-gas. The spent water-gas has thus just sufficient fuel value to compensate for the radiation loss, provided that it be burnt in the most economical manner. One proposal for the economical utilisation of heat from the spent water-gas is to effect its combustion by the reduction of some comparatively readily reducible oxide, such as copper oxide, the heat being recovered by re-oxidation of the hot copper with air. By a suitable arrangement of shells in a generator of the block type, it would thus be possible to reduce the iron oxide in one shell and oxidise the copper in another, burning the whole of the water-gas within the unit. The re-oxidation of the reduced copper with air would, however, probably occasion a very great local elevation of the temperature.

*The purification of hydrogen from the steam-iron process.*

The general methods of purification in use entail merely the removal of the carbon dioxide and hydrogen sulphide.

Sulphur is the most dangerous impurity in hydrogen employed for catalytic purposes; whilst it is customary to remove the hydrogen sulphide from the original water-gas, the organic sulphur compounds such as carbon bisulphide and thiophene remain. Owing to the high percentage of carbon monoxide in the water-gas it would not be feasible to remove this by catalytic decomposition on the surface of hot nickel according to the reaction,



The quantity of carbon deposited from the carbon monoxide would prove a serious factor in the operation of this (the Carpenter-Evans) process unless the operation were conducted at very high temperatures.

It is well known that in the catalytic water-gas process the sulphur compounds are converted into hydrogen sulphide by hydrolysis at the surface of the catalyst,  $\text{CS}_2 + 2\text{H}_2\text{O} = 2\text{H}_2\text{S} + \text{CO}_2$ . In the presence of suitable catalytic agents this reaction proceeds much more rapidly than the hydrolysis of carbon monoxide (the water-gas reaction); thus by admitting a small quantity of steam to the water-gas and passing the mixture over a carefully prepared alumina-iron oxide catalyst, the organic sulphur compounds undergo hydrolysis and but little of the carbon monoxide is converted. Precipitated alumina containing suitable quantities of water will actually hydrolyse carbon bisulphide in the cold. The presence of sulphur compounds in the hydrogen is due chiefly to the formation of sulphides during the reduction period; this can most

conveniently be obviated by working with a water-gas free from sulphur.

Hydrogen sulphide can also be removed from hydrogen by a process of selective combustion. Iron oxide admixed with certain promoters can be utilised for the oxidation of the hydrogen sulphide at a temperature of about 280° C., according to the reaction  $2\text{H}_2\text{S} + \text{O}_2 = 2\text{H}_2\text{O} + \text{S}_2$ . A small quantity of steam must be present to prevent the reduction of the iron oxide. The sulphur is carried forward in the gas stream and sublimes in the colder portions of the tube. The process is, however, complicated by a number of interesting side reactions. If the correct ratio of hydrogen to steam as calculated from the data of Preuner and Chaudron be employed, the following equilibrium is simultaneously obtained:  $3\text{S} + 2\text{H}_2\text{O} = 2\text{H}_2\text{S} + \text{SO}_2$ . Although the equilibrium concentrations of hydrogen sulphide and sulphur dioxide at the temperature of operation are small, they are by no means negligible, and as a result an appreciable quantity of flowers of sulphur is deposited with the condensed water in the condenser. In addition, if the catalyst column be long, rehydrogenation of the sulphur vapour occurs,  $\text{S}_2 + 2\text{H}_2 = 2\text{H}_2\text{S}$ . This phenomenon is well marked in catalysts containing nickel. Still a third possible method for the removal of hydrogen sulphide is its selective oxidation in a liquid medium, e.g. colloidal iron hydroxide, the iron sulphide being regenerated by aeration. Only partial oxidation of the sulphur occurs, the greater bulk being deposited as free sulphur,  $2\text{FeS} + 2\text{H}_2\text{O} + \text{O}_2 = 2\text{Fe}(\text{OH})_2 + \text{S}_2$ .

The sulphides of copper and nickel undergo a similar oxidation, a reaction possibly common to the sulphides of all heavy metals. The reaction velocity at 20° C. varies in a marked manner from metal to metal; nickel is extremely reactive, copper almost inert, and iron of medium activity. It would appear possible that by a suitable choice of metal and the addition of a catalytic oxidation accelerator, e.g. a vanadium salt, a reaction velocity comparable with the rate of formation of the sulphide could be obtained. Under these conditions a purely catalytic process for removing sulphide would become operative as distinct from the discontinuous process now in operation in certain coal-gas plants.

For the removal of carbon monoxide there are likewise several alternative schemes which appear capable of industrial development. Of these may be mentioned the method of selective combustion, the process utilising the water-gas reaction, and that of methanation. The methanation process of Sabatier, in which nickel is used as contact agent for the reaction  $\text{CO} + 3\text{H}_2 = \text{CH}_4 + \text{H}_2\text{O}$ , proceeds rapidly and smoothly at relatively low temperatures, provided that the hydrogen is free from substances containing sulphur. The method has but small merit for hydrogenating processes in which a circulatory system is in use, for apart from the actual hydrogen consumption—a not unimportant item in a gas containing 2–4% of carbon monoxide—the methane rapidly accumulates in the system, lowering the partial pressure of the hydrogen, unless frequently removed by blowing off.

The utilisation of the water-gas reaction appears much more promising. It has already been indicated that in the steaming phase, 7–10 times the amount of steam actually necessary is passed through the retort system. Since the retort contains iron oxide, it follows that the water-gas equilibrium should be obtained in the effluent hydrogen containing but small quantities of carbon monoxide and dioxide. This was actually found to be the case; the ratio of carbon monoxide to dioxide in the hydrogen was found to conform to the ratio of steam to hydrogen as demanded by theory, the ratio being high at the commencement and very low towards the end of a make. It should thus be

possible to lower the carbon monoxide content of the hydrogen, either by passing the gas as it issues from the retort through another retort in the same bank maintained at a somewhat lower temperature, *e.g.* 450° C., or by mixing the hydrogen from which the carbon dioxide has been removed (or even hydrogen which has already passed through the hydrogenation plant, and is consequently rich in carbon monoxide) with the steam entering the system one or two minutes after the commencement of a make. The following figures indicate the purification which could be effected on an average make of hydrogen containing 0.3% CO and 1.8% CO<sub>2</sub> with these alternative methods.

1. Passage of the make through a catalyst chamber maintained at 450° C. with a hydrogen:steam ratio of 1:3.—Entering gas, 0.3% CO, 1.8% CO<sub>2</sub>; effluent gas, 0.07% CO, 2.03% CO<sub>2</sub>.

(2) Passage of the hydrogen after removal of the carbon dioxide through the Lane setting two minutes after commencement of the make, when nearly 50% of the make has already been produced: Entering gas, 0.3% CO, 0.0% CO<sub>2</sub>; effluent gas, 0.13% CO, 0.9% CO<sub>2</sub>. The method of selective combustion involves the removal of the sulphur compounds from the hydrogen. The hydrogen is then mixed with a little more than the requisite amount of oxygen—for very small concentrations of carbon monoxide with twice the theoretical amount for complete combustion—together with a small amount of steam, and passed over suitable catalytic materials such as activated iron or copper oxides. Combustion of the carbon monoxide is complete and a little water is formed. The oxygen is most conveniently generated by electrolysis. This process works admirably on units dealing with 1000 cb. ft. per hour, but has not yet been tested on a larger scale.

#### *The water-gas catalytic process.*

It has already been indicated that the minimum water-gas consumption for the steam-iron processes lies in the neighbourhood of 2.5 volumes per volume of hydrogen, and that the steam consumption is at least 4 volumes, which could theoretically be reduced to about 1.5 volumes per volume of hydrogen.

The development of the Mond and Langer catalytic process by the Badische Co. has resulted in the production of 1 vol. of hydrogen by the consumption of 1.2 vols. of water-gas and 2.5–3 vols. of steam. Apart from the problem of the removal of the carbon dioxide, accomplished automatically in the steam-iron processes, it is much more economical than these latter in operation.

The resulting gas has, however, to be freed from some 30% of carbon dioxide and 2–3% of carbon monoxide; this is generally accomplished by pressure-scrubbing with water and then with aqueous salts.

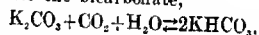
Although the process is more economical than the steam-iron processes, it has not been developed on small-scale work, being in general confined to units so large that the water-gas and steam costs are the sole determining factors. Further investigation on many points is desirable to stimulate more rapid extension of the process.

Water-gas producers for the production of blue gas suitable for direct use in a hydrogen converter will have to be designed. Of the usual 5–6% of inert nitrogen and methane present—sufficient to condemn the hydrogen for many industrial purposes, unless the cost of production were very low—at least half and frequently much more than half is derived from the air and is not due to the presence of nitrogenous substances in the coke. The faulty average after the air-blow is very clearly marked if the nitrogen content of the resulting gas be plotted against the period of the make. Again, although the water-gas reaction is exothermic ( $\text{CO} + \text{H}_2\text{O} = \text{H}_2 + \text{CO}_2$ , +10,000 cal.), the amount of heat liberated

is usually insufficient in small units to maintain the system at the usual temperature of operation, 550°–600° C., even with good heat-interchangers and lagging; the loss by radiation is extremely great. This problem would be solved in the most suitable manner by the utilisation of a catalyst operative continuously at 450° C. instead of 550°–600° C. Under these conditions the reaction would be autothermal and a much purer gas would be obtained, since the value of *K*, the equilibrium constant of the water-gas reaction, sinks from 0.24 at 550° C. to 0.10 at 450° C. With a steam:gas ratio of 2.5:1 the carbon monoxide content would be 3.0% at the higher and 1.17% at the lower temperature. Although many of the activated catalysts will operate on purified water gas at this temperature for reasonable periods, yet up to the present time no one of them has stood the test of operation under industrial conditions.

For small plants alternative methods to pressure scrubbing for the removal of the carbon dioxide and monoxide suggest themselves. Electrolytic regeneration of caustic soda from sodium carbonate would prove a simple but slightly expensive method of operation. By the employment of a divided cell, the electrolytic hydrogen produced could be utilised to augment the main supply. The consumption of water-gas would be about 1200 cb. ft. and of power about 160 kw. per 2000 cb. ft. of hydrogen.

It would be more economical to absorb the carbon dioxide in sodium or potassium carbonate and then to regenerate the bicarbonate,



To effect complete dissociation into the normal carbonate it would be necessary to heat the salt to about 100° C. and to remove the carbon dioxide by vacuum pumping or by spraying in a current of air or steam if the carbonate be in the form of a solution. Absorption would be effected at ordinary temperatures where the partial pressure of the dioxide is very low. The approximate theoretical fuel consumption for decomposition and heating of the bicarbonate to 100° C. is 9700 B.Th.U. per 1000 cb. ft. of scrubbed hydrogen, equivalent to a fuel consumption of only 33 cb. ft. of water-gas. Even if the dissociation process operated with a thermal efficiency of only 10%, this would only entail a consumption of 0.3 vol. of water-gas per volume of hydrogen produced. Where hydrogen is being manufactured for the synthesis of ammonia absorption in ammonia could be employed. The various methods for the removal of carbon monoxide have already been discussed.

#### *The direct production of hydrogen from coke.*

The hydrocarbons readily decomposed at low temperatures, such as acetylene or ethylene, are too valuable to be used as a source of hydrogen in competition with the methods already discussed. Other hydrocarbons, such as methane and the paraffins, require such a high temperature for their dissociation as to render their technical utilisation a matter of some difficulty; thus to produce hydrogen containing less than 2.5% of methane a temperature of over 900° C. would be necessary.

The methods based upon the reactions of the water-gas producer are more promising:—

- (i.)  $\text{C} + \text{H}_2\text{O} = \text{CO} + \text{H}_2$ .
- (ii.)  $\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{CO}_2 + \text{H}_2$ .
- (iii.)  $\text{C} + \text{CO}_2 \rightleftharpoons 2\text{CO}$ .

For the ultimate conversion of carbon and steam into hydrogen and carbon dioxide by means of these reactions it is necessary to operate at a low temperature. As has already been noted, it is quite possible to arrive at an equilibrium in the water-gas reaction (ii.) at temperatures as low as 400° C. with the aid of a catalyst. If some means were at hand for accelerating (i.) the most economical solution

of the problem of hydrogen manufacture would be found.

The reaction is heterogeneous and catalytic stimulation is consequently difficult. Several methods for increasing the reaction velocity have, however, been suggested, and some of them have proved feasible on a semi-industrial scale. Thus Harries (J. Gasbeleucht., 1894, 81) utilising a very reactive form of carbon, viz. wood charcoal, obtained equilibrium without the addition of any catalytic material at a temperature of 700° C., but apart from the necessity of employing a special fuel the reaction velocity was found to be exceedingly slow.

Bergius (G.P., 259,030/11, 262,831/12) showed that reactions (i.) and (ii.) could be accelerated by the simultaneous employment of pressure and a catalyst. He was able to obtain a reaction velocity sufficiently high for technical utilisation at a temperature of 300° C. The pressure was maintained above the critical pressure of water (89 atm.), whilst chlorides, and especially thallium chloride, were employed as catalytic materials. The gas so obtained contained but small traces of hydrocarbons.

Attempts have likewise been made to run the water-gas producer at low temperatures, e.g., 500°–800° C., and to accelerate the reactions (i.) and (ii.) by the addition of suitable catalytic materials. Haber suggested that the ash, and especially the ashes containing iron, accelerated the water-gas reaction and facilitated the interaction between the carbon and steam, but did not influence the reaction velocity of the interaction between carbon and carbon dioxide. This hypothesis was in part confirmed by Gwosdzy (Z. angew. Chem. 1918, 137), who showed that the reaction velocity of the water-gas reaction was greatly affected by the ash content of the coke. A coke containing 8.5% of ash yielded 29% of carbon dioxide, whilst pure carbon (lampblack, 0.1% ash) yielded only 8.6% of carbon dioxide at 560° C. More recent patents describe the spraying of the coke with suitable catalytic materials before feeding into the water-gas generator. The difficulty associated with the process if the ordinary type of generator be employed would largely depend on the nature of the clinker and the mechanism for its removal. For water-gas generators operating at high temperatures, e.g. 1300° C., any increase in constituents forming clinker or fusible slag is to be avoided, but at 600°–700° C. a small increase in clinker content could be permitted. Again the process would appear capable of inversion: powdered coke sprayed with catalytic material could be injected into a current of steam superheated at 700° C., thus obtaining a simultaneous increase in the reaction velocity due to an increase in reacting surface and the elimination of the clinker problem.

Chemical Laboratories, The University,  
Cambridge.

## THE ABSORPTION OF ETHYLENE AND PROPYLENE BY SULPHURIC ACID.

BY SYDNEY GLENN PRESTON PLANT AND NEVIL VINCENT SIDGWICK.

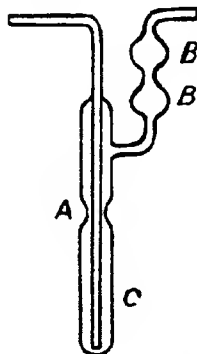
It has been known for a long time that ethylene can be absorbed by sulphuric acid, and this was made the basis for the commercial production of ether by Fritzsche (Chem. Ind., 1898, 21, 27; 1912, 35, 637). The dissociation and decomposition of ethylsulphuric acid at different temperatures has been investigated by Kremann (Monatsb., 1910, 31, 211). The object of the present work was to investi-

gate the effect of varying the conditions upon the rate of absorption of ethylene by sulphuric acid, and also to study the products and the mechanism of the reaction. The ethylene used was prepared by passing the vapour of 97% alcohol over pumice soaked in phosphoric acid and maintained at a temperature of 280°–300° C. The gas was passed from a holder through two wash-bottles containing cold concentrated sulphuric acid, and then through the absorption vessels in series immersed in an oil thermostat. After leaving the last vessel, the gas was passed through another sulphuric acid wash-bottle and collected in a second holder. In this way pressure was applied at the beginning of the series and suction at the end, so that it was possible to maintain a fairly regular stream of gas through as many as five absorption vessels in addition to the wash-bottles for about three hours. The vessels were removed after definite intervals of time, washed with ether and then with water, and finally dried and weighed. The gas was repeatedly analysed by absorbing the ethylene in bromine water, and the volume sent through the vessels during each stage was read off from the graduations on the gas-holder. The gas in the second holder was transferred to the original one and the absorption continued. When not connected up in the series the vessels were kept in the desiccator, the delivery and exit tubes being closed.

### The absorption vessel.

Since the experiments were carried out by having an acid of different strength in each of the vessels in the thermostat, it was necessary for the conditions in each to be comparable. By passing the gas at such a rate that the quantity absorbed is small compared with that sent through, the amount passing through each vessel will be approximately the same. Experiments have shown that above a certain minimum value the speed of the gas has only a small effect upon the absorption. The vessels were tested by filling each with sulphuric acid of the same strength and passing the gas for a definite time through all in series, and then calculating the percentage increase in weight of each acid. If the vessels are satisfactory the results would be almost identical. Several variations of the ordinary type of Liebig bulbs were tested, but these proved to be unsatisfactory owing to the difficulty of controlling the distribution of the acid in the bulbs. These difficulties were overcome by using the absorber represented in Fig. 1.

The corresponding parts of each vessel were made from tubing of the same bore, the delivery tube being sealed in a central position with its end as near the bottom as possible. The constriction, A, served to break the bubbles which rose during the passing of the gas, and thereby to check the tendency of the liquid to be carried over into the safety bulbs, BB. The liquid was carried over to a small extent in the final stages of the absorption into the lower bulb, and on a few occasions into the upper one. A small mark, C, was made on the outside of each vessel at a point so as to give as nearly as possible the same amount of liquid in each, and they were filled up to this point for each absorption. Five vessels were made and tested by the method described and found to be satisfactory.





*The influence of the speed of the gas upon the absorption.*

To investigate this point the gas used in each absorption was taken from a large storage gas-holder, so that the percentage of ethylene in it was constant. It was passed from a graduated bottle of five litres capacity through two wash-bottles containing cold strong sulphuric acid, and then through one absorption vessel containing in every case 99.3% acid and immersed in the thermostat at 70° C. The gas leaving the final wash-bottle was allowed to escape into the outer atmosphere, thereby avoiding changes of pressure which a receiving gas-holder introduces. The pressure driving the gas through the vessel was maintained almost constant by allowing water to siphon from an elevated reservoir into the gas-container, keeping the level of the water in the reservoir carefully regulated. Before each absorption the gas was allowed to bubble for some time through the first two sulphuric acid wash-bottles in order to remove all the air. Before introducing the absorption vessel the gas in the container was brought to atmospheric pressure and its volume read. After each absorption, which was of two hours' duration, the gas in the container was again brought to atmospheric pressure and its volume observed. The following results were obtained:—

Speed of gas in litres per hour.	Increase in weight of acid %
0.80 .. ..	1.90
0.74 .. ..	2.09
1.07 .. ..	2.50
1.54 .. ..	2.90
2.29 .. ..	3.07
3.81 .. ..	3.43

These figures indicate that above 1.7 litres per hour at 70° C. the effect of small variations in the speed of the gas is quite small. This result is quite empirical and will depend upon the nature of the absorption vessel used, since upon this depends the relative amount of liquid surface exposed to the gas and the extent of the stirring.

*The effect of the purity of the ethylene upon the rate of absorption.*

This was investigated by passing mixtures of ethylene and air of different compositions through 99.3% acid at 70° C. for 2½ hours and observing the percentage increase in weight in each case. The following are the results obtained:—

Ethylene %	Increase %	Ethylene %	Increase %
92.7 .. ..	3.66	50.7 .. ..	2.62
86.4 .. ..	3.37	46.4 .. ..	2.30
84.0 .. ..	3.53	41.1 .. ..	2.21
78.9 .. ..	3.62	37.3 .. ..	1.91
72.7 .. ..	3.53	28.6 .. ..	1.44
69.6 .. ..	3.52	25.2 .. ..	1.32
64.6 .. ..	3.41	17.9 .. ..	1.06
59.8 .. ..	3.14	6.0 .. ..	0.48
57.3 .. ..	2.72		

It follows from these figures that above about 69% the percentage of ethylene in the gas has little effect upon the degree of absorption. Confirmation of these results is obtained from the absorption curves.

*Analysis of the product of the absorption.*

Upon pouring the product of the absorption into water an oil separated at the bottom of the vessel and proved to be diethyl sulphate. Ethylsulphuric acid, some free sulphuric acid, and water are also present in the product. To estimate the amounts of each present the acid liquid was extracted twice by shaking with light petroleum spirit (b.p. 40°–60° C.), and the ethylsulphuric acid and diethyl sulphate estimated by oxidation with a standard solution of potassium bichromate (Benedict and Norris, Amer. Chem. Soc., 1898, 20, 293). The diethyl

sulphate remaining with the petroleum spirit was hydrolysed by heating with water for ½ hour before estimating it. As a rule the difference between the sum of the amounts of ethylene corresponding to each product as found by analysis and the increase in weight of the acids during the absorption did not exceed 2%. The amounts of free sulphuric acid and water present in the product were calculated from a knowledge of the original quantity of acid and its strength. A trace of free alcohol is present in the product as a result of hydrolysis, but will appear as ethylsulphuric acid in the analysis. The actual quantity present is very small, and certainly less than the error (usually about 2%) appertaining to the analytical methods, so that any estimation of it would have been of little value.

*The absorption at 50° C.*

Three vessels were arranged in series, each containing an acid of different strength, the weakest acid being the first and the strongest the last of the series. The following figures show the percentage increase in weight of the acids based upon the actual (anhydrous) sulphuric acid present. The experiment was conducted in stages of 2–3 hours each, the vessels being weighed at the end of each interval. No appreciable decomposition took place, the acids remaining quite clear throughout, and becoming very slightly brown towards the end.

Time in hours.	Percentage increase in weight.		
	93.1% acid.	97.6% acid.	99.3% acid.
3 .. ..	1.24 ..	1.70 ..	2.52
6 .. ..	2.48 ..	3.57 ..	5.66
9 .. ..	3.63 ..	5.49 ..	9.38
11 .. ..	4.82 ..	7.15 ..	12.98
15 .. ..	6.39 ..	10.90 ..	21.63
18 .. ..	7.99 ..	14.79 ..	26.78
21 .. ..	9.68 ..	19.10 ..	29.53
23.5 .. ..	11.10 ..	22.21 ..	30.99
26 .. ..	12.51 ..	24.49 ..	32.01
28.5 .. ..	13.72 ..	26.16 ..	—
31 .. ..	14.73 ..	27.98 ..	—
33 .. ..	15.44 ..	28.10 ..	—
35 .. ..	16.03 ..	28.67 ..	—
37 .. ..	16.56 ..	29.23 ..	—

These results are plotted as curves in Fig. 2.

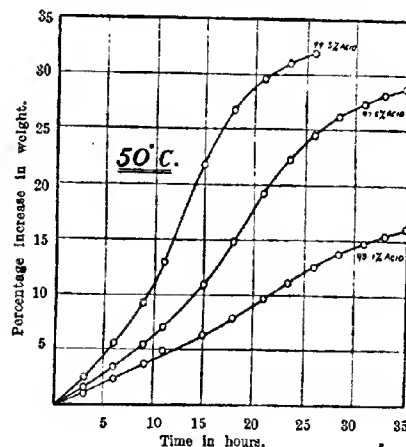


FIG. 2.

The following are the results obtained from the analysis of the products:—

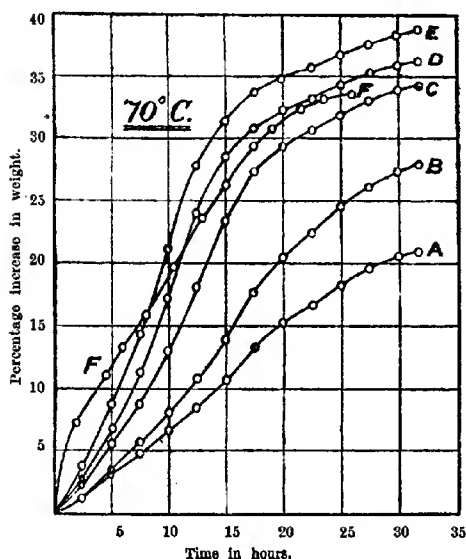
Acid strength.	Increase in weight, %	Ethylsulphuric acid, %	Diethyl sulphate, %	Sulphuric acid, %	Water, %
93.1 ..	16.66 ..	60.1 ..	Nil ..	33.9 ..	6.0
97.6 ..	29.23 ..	73.9 ..	15.8 ..	8.4 ..	1.9
99.3 ..	32.01 ..	72.7 ..	22.2 ..	4.6 ..	0.5

*The absorption at 70° C.*

These experiments were carried out in the same manner as those at 50° C., with the following results:—

Time in hours.	Percentage increase in weight.				
	93.1% acid.	95.8% acid.	98.1% acid.	98.8% acid.	100.1% acid.
2.5	1.36	1.28	2.27	2.75	3.83
5	3.16	3.52	5.59	6.82	8.89
7.5	4.73	5.87	8.94	11.32	14.29
10	6.59	8.05	13.05	17.16	21.11
12.5	8.62	10.84	18.11	23.93	27.79
15	10.59	13.95	23.32	28.50	31.46
17.5	13.29	17.78	27.31	30.97	33.64
20	15.22	20.47	29.36	32.37	34.97
22.5	18.56	22.37	30.55	33.20	35.69
25	18.19	24.49	31.95	34.39	36.75
27.5	19.52	28.01	33.04	35.23	37.58
30	20.50	27.30	33.89	35.93	38.24
31.75	20.63	27.98	34.25	36.23	38.62

These results are plotted as curves in Fig. 3.



A, 93.1% acid; B, 85.8%; C, 98.1%; D, 98.8%; E, 100.1%; F, 14% "oleum."

FIG. 3.

There are two small irregularities in each of these curves. The third point in each case, corresponding to 7½ hours, is slightly lower than would be expected from the general direction of the curves; in this case it was found that the ethylene had been passed at too slow a rate. The second and more obvious irregularity is at the ninth point after 22½ hours, which is again too low, but in this case it is due to the fact that the percentage of ethylene in the gas fell to 61.2%. Apart from these two points each curve is quite regular, showing at first a continuous acceleration in the rate of absorption followed by a retardation as the reaction proceeds. The colour of the acid gradually becomes yellow. In no case, however, was there any appreciable decomposition, even with the maximum absorption obtained, all the liquids remaining quite clear. The possibility of the decomposition of the ethylsulphuric acid was investigated by inserting after the absorption vessels a vessel containing about 2 c.c. of *N*/10 iodine solution diluted with water. In no case was there any appreciable decolorisation of the iodine. The absorption was stopped after 31½ hours, at which point it became difficult to maintain a steady stream of gas through the vessels owing to the large bulk of liquid in them. The volume of liquid increases about 100% during the absorption.

The following table gives the composition of the products obtained:—

I. Acid strength, %	II. Increase, %	III. $C_2H_5.H_2SO_4$	IV. $(C_2H_5)_2SO_4$	V. $H_2SO_4$	VI. $H_2O$
93.1	20.83	69.5	2.2	22.5	4.8
95.8	27.08	79.1	9.4	8.2	3.3
98.1	34.25	74.8	20.3	3.5	1.4
98.8	36.23	68.1	29.3	1.7	0.9
100.1	38.62	70.0	30.0	nil	nil

*The absorption at 100° C.*

Three of the acids used at 70° (93.1%, 98.1%, and 100.1%) were placed in the same vessels and the absorption investigated in the same manner at 100° C. The following are the figures obtained:—

Time in hours.	Percentage increase in weight.		
	93.1% acid.	98.1% acid.	100.1% acid.
2.5	2.43	5.18	7.43
4	4.34	8.88	11.86
5.5	6.37	13.08	16.67
7.5	8.63	17.11	21.35
9	10.23	20.14	24.52
11	12.39	24.12	28.28
13	14.80	27.02	30.72
15	16.88	28.44	32.68
17	18.09	29.22	32.97
19	19.70	29.34	33.72
21	20.36	29.34	34.00

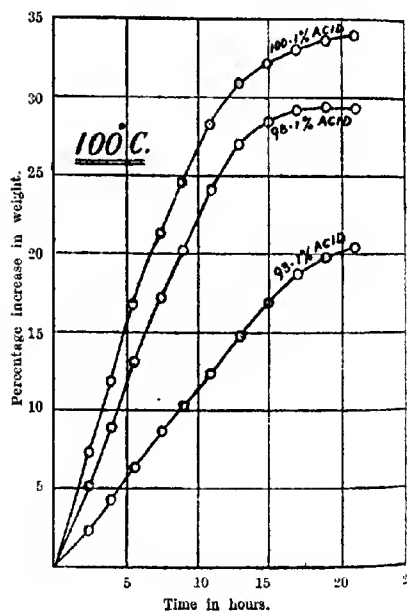


FIG. 4.

These curves (Fig. 4) show the same regularity which characterises those at 70° C. Decomposition of the ethylsulphuric acid is quite marked at 100° C. After running for 1½ hours the strongest acid showed a considerable blackening in colour, and after two hours this was very marked. At the end of 2½ hours the 98.1% acid was brownish yellow and the weakest acid slightly yellow. All the acids were quite black after 11 hours. The question of decomposition has been more fully investigated at 125° C.

The analysis of the products was difficult owing to the decomposition which had taken place interfering with the ease and rapidity of the separation with petroleum spirit. The results obtained were of little value and are omitted.

*The absorption at 125° C.*

This was investigated mainly for the purpose of observing the effects of the decomposition of ethylsulphuric acid. The gas was more rapidly absorbed at this temperature than at 100° C. until the decomposition of the product retarded the increase in weight and finally made it negative. Only two acids were used, viz.: 93.1% and 100.1%. Both rapidly became black and opaque, and an analysis of the products was impossible. The stronger acid became almost solid from the quantity of carbonaceous material which separated. The following figures were obtained:—

Time in hours.	Percentage Increase.	
	93.1% acid.	100.1% acid.
1.5	2.26	5.41
2.5	3.63	8.92
4	5.53	7.81
5.5	6.46	7.12
7	7.80	6.29
8.5	8.29	4.39
10	7.71	2.65
11.5	6.19	—
13	4.33	—
14.5	2.56	—
16	-0.31	—

*The absorption by oleum at 70° C.*

The absorption of ethylene by fuming sulphuric acid was investigated by passing the gas through 1% oleum contained in one of the absorption vessels at 70° C. The following figures indicate the mechanism of the reaction is essentially different from all the other cases:—

Time in hours.	Percent. inc. in weight.	Time in hours.	Percent. inc. in weight.
2	7.47	15	26.25
4.5	11.09	17.5	29.48
6	13.29	19	30.86
8	15.90	21.5	32.56
10.5	19.68	23.5	33.11
13	23.53	26	33.54

From a study of the curve given in Fig. 3 it is at once apparent that the autocatalytic character, which is so well marked with the other acids, is absent. The absorption in the earlier stages is very rapid, and this is in all probability due to the combination of the free sulphur trioxide with the gas to give carbyl sulphate, the anhydride of ethionic acid. After a few hours the absorption is proceeding at a rate which corresponds to that which would be observed with about 98% acid, which indicates that the carbyl sulphate acts merely as a diluent in the same manner as water.

*The mechanism of the reaction.*

Apart from decomposition there are three successive stages in the absorption, viz.: (1) The solution of ethylene in the liquid, (2) its reaction with sulphuric acid to give ethylsulphuric acid, and (3) its reaction with ethylsulphuric acid to give diethyl sulphate. It is not necessary for all the sulphuric acid to be converted into ethylsulphuric acid before reaction (3) proceeds, as the analyses prove. The theoretical increase in weight for all the acid to be converted into ethylsulphuric acid is 28.57%. At this point, however, a considerable quantity of diethyl sulphate has been formed. The quantity of ethylene actually dissolved as such in the acid being no doubt negligible, the observed increase in weight is a measure of the amounts of reaction (2) and (3) which have taken place. The rate at which the gas was passed was such that a small change in its speed had no appreciable effect upon the absorption, so that the liquid phase must be regarded as saturated with ethylene. These two reactions proceed at rates which are proportional to the products of the concentration of ethylene and the concentrations of sulphuric acid and ethylsulphuric acid respectively.

The curves indicate that the ethylsulphuric acid has a distinct autocatalytic effect, which can also be seen by subtracting each figure from the one succeeding it for any absorption with the exception of those into which decomposition enters. This can be explained by the greater solubility of ethylene in the liquid phase as ethylsulphuric acid accumulates in the system, which is what one would expect. At the end of the experiment, where the rate of absorption is very much diminished, there is still about 70% of the primary product present, so that ethylene must combine with ethylsulphuric acid more slowly than with sulphuric acid.

*The amount of diethyl sulphate present at different stages in the absorption.*

This question, which has been previously referred to, was more fully investigated by passing the gas through three vessels, each containing 98.1% acid, immersed in series in the thermostat at 70° C. and by taking out the vessels at convenient points in the absorption and analysing the products in the usual manner after observing the percentage increase in weight. The following are the results obtained:—

Increase in weight, %	$(C_2H_5)_2SO_4$	
25.14	..	7.9
29.37	..	14.5
32.42	..	21.1

Diethyl sulphate begins to appear after about a 20% absorption. The effect of varying the different conditions upon the production of diethyl sulphate can be investigated by a study of the absorption curves together with the corresponding analyses, and a comparison of the results so obtained with this standard curve for 98.1% acid at 70° C. The influence of the water present with the sulphuric acid is found to be small apart from its retarding action upon the absorption in general. The amount of diethyl sulphate present at any given degree of absorption is practically independent of the strength of the acid used.

*The absorption of propylene by sulphuric acid.*

Experiments have been carried out with propylene on lines similar to those with ethylene. It has been recognised for some considerable time that the former gas was far more readily absorbed by sulphuric acid than the latter. This has been confirmed. It was found that when the gas was passed through 97% sulphuric acid at 25° C. a 50.06% increase in weight occurred after two hours. Absorptions were conducted using acids of strengths between 80% and 90% at 25° C., and the velocities measured were of the same order as those obtained with ethylene and 100% acid at 70° C. The chemical reactions which take place in the case of propylene are far more complex than those between ethylene and sulphuric acid. Apart from the production of propylsulphuric acid and dipropyl sulphate, a colourless oil separated out on the surface of the acid during the course of the absorption. This was separated, washed with an aqueous solution of sodium carbonate, and dried over calcium chloride. The liquid proved to be a mixture, and on distillation under a pressure of 30 mm. the boiling point gradually rose from 55° to 155° C. An investigation showed that this product contained no sulphur, and, furthermore, appeared to consist of saturated compounds. Three fractions were collected on distillation, analyses were made, and various physical constants determined. It was found that some oxygen was present and that the liquid reacted with metallic sodium. It seems probable that the colourless liquid which separates out during the absorption of propylene by sulphuric acid is a mixture of open-chain secondary alcohols. These have probably been produced by propylene

polymerising to give a higher olefine with the double bond in the 1.2-position, having a formula  $\text{CH}_3(\text{CH}_2)_{10}\text{CH}:\text{CH}_2$ . Sulphuric acid then adds on to the double bond and the compound so produced is hydrolysed to give the corresponding secondary alcohol. The physical constants indicated that the complexity of the molecule was of the order of  $\text{C}_{12}$ .

Organic Chemistry Laboratory, Oxford.

#### PRELIMINARY NOTE ON THE ABSORPTION OF LIGHT BY CAOUTCHOUC.

S. JUDD LEWIS, D.S.C., F.I.C., AND B. D. PORRITT, M.S.C., F.I.C.

In view of the changes which are produced in rubber when exposed to light and air, it is somewhat surprising that no record appears to exist of any effort having been made to study the action of light on caoutchouc in a quantitative manner. It was therefore decided to carry out some preliminary experiments to determine the character of the absorption of light of short-wave length by caoutchouc, to which experience has shown that the successive physical and chemical changes which occur during "perishing" must be attributed.

With this object a specially good sample of "fine hard Para" rubber was selected in the crude condition before subjection to any manufacturing operation. This was cut into fine strips and submitted to repeated extraction with cold distilled water for a period of over a week, followed by extraction with cold acetone until the washings were no longer coloured. A final prolonged digestion with several changes of absolute alcohol was employed to ensure the complete removal of resins and soluble colouring matters, and the extracted rubber was thereafter dried at ordinary temperature in a current of hydrogen to prevent oxidation. The dry material was then transferred to a stoppered separating funnel in which it was allowed to swell and slowly dissolve in anhydrous ethyl ether (purified by treatment with sodium and fractional distillation) without agitation. The clear caoutchouc solution was withdrawn at intervals before it became unduly viscous, from the bottom tap, the passage of proteid and insoluble matter being prevented by the insertion of a small plug of asbestos fibre, and the volume removed was replaced by the addition of fresh solvent.

In this way a sufficient quantity of a perfectly transparent, colourless solution of caoutchouc was obtained containing approximately 0.94% of solute by weight. As it was found that this solution gave only very feeble absorption, it was reduced by spontaneous evaporation to about two-thirds of its original volume, thus increasing its strength to approximately 1.5 per cent.

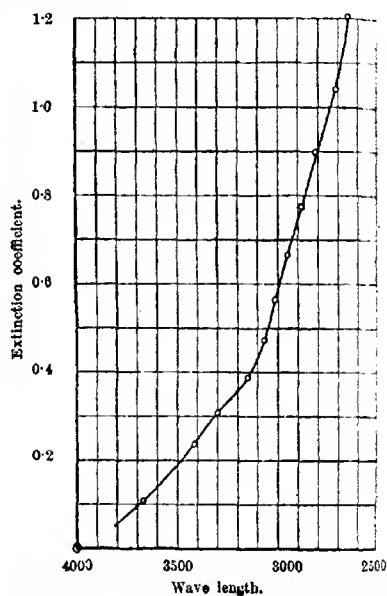
The method of procedure followed was similar to that described elsewhere (Proc. Roy. Soc. B., 89, 329), with the exception of the photometer employed, which was of the new sector type, the details of which have lately been published (Chem. Soc. Trans., 1919, 115, 312).

The 2 cm. cell containing the solution was placed in the one path of light, and a duplicate cell filled with the purified ether, similar to that used for the preparation of the solution, was placed in the other beam for the purpose of providing a control. The absorption effect was therefore confined to the solute alone, that of the solvent being eliminated. The ether employed was found to give no absorp-

tion at wave lengths above 2170 sufficient to interfere with the experiment.

The results obtained are exhibited in the accompanying curve, in which are plotted the values of the extinction coefficient, that is of  $\log I/I'$ , relative to a 3% solution in a cell of unit thickness as ordinates against wave lengths as abscissae ( $I$  is the intensity of the incidental light, and  $I'$  is that of the light transmitted). The curve has not been smoothed out, as the slight irregularities may prove to be significant.

In considering these preliminary results it is in the first place remarkable that caoutchouc should be so transparent to light as to call for the use of so concentrated a solution as 1.5% in a 2 cm. observation tube.



Ultra-violet absorption spectrum curve of ethereal solution of caoutchouc calculated on a 3% solution in a 1 cm. cell.

The curve exhibits only a general absorption which is fairly strong for wave-lengths below 2700, but rapidly diminishes with increase in wave-length.

Neither with the strong solution nor with weaker ones has there been any decisive evidence of absorption bands, but the investigation has not gone far enough to say whether caoutchouc is capable of exhibiting these or not.

Further work is in progress on different types of rubber and allied compounds with a view to confirming these preliminary observations, and if possible securing some evidence regarding the constitution of caoutchouc.

The authors desire to thank the North British Rubber Co., Ltd., for their permission to publish this work, some of which was carried out in their research laboratory.

Staple Inn Buildings, London.

Rubber Research Association,  
University College, London.

MR. F. H. CARR IN THE CHAIR.

BY SAMUEL COFFEY, B.SC. (LOND.).

Linseed oil was heated at 250° in sealed tubes which contained very little air, so that oxidation effects and loss of volatile matter were eliminated. The same sample of "pure" raw oil, direct from the presses, was used throughout the experiments, and as it was obtained from a reputable firm especially for the work, it was assumed to be unadulterated, and purification was deemed unnecessary. Its constants were: Sp. gr. at 20° C., 0.9310; saponification value, 189.0; acid value, 6.4; iodine value, 189.1; hexahromide value, 43.0; glycerol. 0.0%. At the commencement of the experiment two similar tubes containing approximately equal quantities of oil were sealed and heated in a sand bath in an air oven to 250°-260° C.; a similar tube at longer, containing the same amount of oil, open to the atmosphere, and in which a thermometer was placed, was also inserted in the sand. In no case as the temperature allowed to rise above 260°

The original oil was of a clear golden brown colour, which after seven hours' heating in the sealed tube had changed to a very pale yellow with a slight green fluorescence, which, according to Lewkowitch, is characteristic of "litho oils" (linseed and tung oils, which have been heated without appreciable oxidation). Further heating appeared to have only a slight effect on colour; even after 42 hours' heating the colour and the fluorescence were not very much more intense than those of the oil heated for seven hours, and in each case the product was paler in colour than the original oil. The specific gravity at 20° C. (determined by the density bottle method) and the relative viscosity at the same temperature (determined by Poiseuille's capillary tube method; original oil = 1) are given in Table I.

Time of heating, hrs.	Sp. gr. at 20° C.	Relative viscosity at 20° C.
0	0.9310	1.00
1	0.9480	2.28
13.5	0.9634	8.52
21	0.9714	4.07
28	0.9728	17.50
35	0.9810	34.40
42	0.9875	49.90

Further qualitative evidence is obtained from a study of the relative solubilities of these oils. In ether and light petroleum, in each case solution was complete, but more solvent was required as the heating continued. In acetone, however, a marked difference was observed. The original oil and the samples which had been heated for 7 and 14 hrs. were miscible with the solvent, but of the oil which had been heated for 21 hrs. a small quantity (about 5%) remained undissolved even after repeated extraction. As the heating continued this insoluble portion became larger; after 28 hrs. it was about 25%, while after 42 hrs. it constituted the larger part of the product. This insoluble product was a thick heavy oil. It is remarkable that the insoluble product should appear after 21 hrs. heating, at the same time as discontinuities in the viscosity, sp. gr., and acid value curves (*cf. infra*) show themselves, and it seems safe to presume that these marked changes are caused by the production of this thick insoluble product which in all probability is identical with that obtained by R. S. Morrell by thickening the oil at 260°—280° C. (J., 1915, 105, *loc. cit.*)

*Chemical properties.*—The acid value and saponification values were determined by titration with

alcoholic potash,\* the hexabromide value by the Hehner-Mitchell method as used by Ingle (*loc. cit.*), and the iodine value by Wijs' method (Lewkowitsch, "Analysis and Technology of Oils, Fats, and Waxes," p. 240 *et. seq.*). It was found by Morrell (*loc. cit.*) that the iodine values of thickened linseed oil determined by this method increased with the time the thickened oil was in contact with the iodine solution. To eliminate this effect as much as possible the oil was left in contact with the solution for exactly 3 hrs. before titration with standard thiosulphate solution. The results of these determinations are given in Table II.:—

TABLE II.

Time of heating, hrs.	Acid value.	Saponif. value.	Iodine value.	Hexabromide value.
0	6.4	189.0	189.1	43.0
7	10.4	188.2	176.7	micro trace
13.5	11.4	188.3	162.1	—
21	12.6	188.7	142.0	—
28.	14.6	189.0	125.2	—
35	15.0	189.0	110.4	—
42	15.0	188.8	107.0	—

These figures show that the saponification value, as in Krumhaar's experiments (*loc. cit.*), remains unaltered. The iodine values decrease fairly regularly as the heating is continued, the decrease being most rapid at first, but there are no breaks on the curve.

The hexabromide value of 43% in the original oil had become almost zero after 7 hrs.' heating, showing that all traces of the most highly unsaturated glycerides had disappeared, probably owing to polymerisation into glycerides of more saturated diacids, e.g.,  $\text{COOH}\cdot\text{C}_{17}\text{H}_{33}\cdot\text{C}_{17}\text{H}_{33}\cdot\text{COOH}$  etc. (*Cf. Morrell, loc. cit.*)

The acid values are particularly interesting. The original oil had an acid value of 6.4.† On heating, however, the value gradually rose in a continuous manner until after 21 hours' heating. The continuity of the curve is then broken, and the acid value rises abruptly to a constant value from 28 hrs. onwards, equal to about 2.4 times its original value.

It is noteworthy that in these experiments the increase in acid value is not nearly so large as when the oil is heated under atmospheric pressure and volatile matter allowed to escape. In Krumhaar's experiments, for example, after heating for 20 hrs. at 300° the acid value was 40 times the original, and showed no indication of becoming constant.

No explanation of this pronounced increase in acid value appears to have been put forward. While appreciating the fact that considerably more experimental evidence is required before the point can be settled, the author would suggest that this is due to the direct hydrolysis of the glycerides by water. Raw linseed oil always contains small amounts of water, hence some hydrolysis will take place, and an equilibrium will be set up according to the mass action law as  $\{\text{C(glyceride)}\} \times \text{C}(\text{H}_2\text{O}) \div \{\text{C(glycerol)}\} \times \text{C(acid)} = \text{K}(\text{constant})$ , and the final acid value for a given oil at constant temperature will be constant as found experimentally. Assuming that K also is small, then for the present case 0.4% of water would be quite sufficient to account for the increase in acid value.

\* Some workers consider alcoholic potash to be unsuitable for determining acid values, but the author has found that with neutral alcohol and well kept solutions identical results are obtained with baryta and with potash. It has also been found that acid and saponification values determined by alcoholic potash gave glycerol values identical with those found by the acetin method, although the acid value was as high as 5.3.

† The allowable acid value for linseed oil seems to be somewhat vague; some authorities give it as up to 6 or 7, whilst some say it should not exceed 1 or 2. It should be remembered, however, that in this case a raw oil was used which had undergone no purifying treatment. For four such oils of widely different origin the author has found values of 5.2, 6.4, 5.2, and 2.0—all above 2 but below 7.

In the case of previous experiments, done under ordinary pressures at temperatures about 300°, the glycerol produced by hydrolysis would be volatilised, and so the hydrolysis would go to completion. Also at these temperatures the glycerol would probably be converted into acrolein and water, and so the latter would be continuously formed, and this would account for the high acid values observed.

In conclusion, the author desires to express his thanks to Prof. F. S. Kipping, D.Sc., F.R.S., for his kind interest and encouragement during this work.

Chemical Dept., University College,  
Nottingham.

## Communications.

### THE DETERMINATION OF AROMATIC HYDROCARBONS IN MIXTURES OF HYDROCARBONS.

BY H. T. TIZARD AND A. O. MARSHALL.

In the course of a general research on the behaviour of fuels in internal combustion engines, it became necessary to devise a simple and accurate method for determining the proportion of aromatic hydrocarbons in petrol. The method outlined in this paper is a development of that described by F. B. Thole (J., 1919, 39 r), according to which the mixture is sulphonated with 98% sulphuric acid and the densities before and after sulphonation determined. Thole claims that by the use of 98% acid aromatic compounds are completely removed, while other saturated hydrocarbons are only attacked to a limited extent. He estimates the separate hydrocarbons benzene, toluene, xylene, by fractionating the sample of petrol and dividing the product into three portions: (a) b.p. 40°–95°, (b) 95°–122°, (c) 122°–150° C. It is assumed that (a) contains all the benzene and (b) all the toluene. The three fractions are then sulphonated separately, and the densities before and after sulphonation determined. The aromatic content is calculated from the difference in densities, making an allowance for the expansion known to occur when aromatic and non-aromatic hydrocarbons are mixed.

Our experiments support Thole's conclusion as to the efficacy of 98% sulphuric acid, and in general confirm the accuracy of his "cutting points," with certain exceptions that will be mentioned later. But the procedure of determining densities before and after sulphonation is open to the great objection that the time involved and care necessary for really accurate density determinations are considerable, and further that the correction to be applied for the divergence of densities of petrols containing aromatic compounds from a simple mixture law is uncertain. Thole's method, too, is only applicable to a very limited extent to the determination of xylene content, since the densities of the three xylenes are different, and it also does not lend itself to a quick and accurate determination of total aromatic content for a similar reason.

The measurement of the critical solution temperature of hydrocarbons in aniline can be used instead of the measurement of density, to determine aromatic content, with greater ease and greater accuracy. Chavanne and Simon, who have worked on similar lines, have recently pointed out (*Comptes rend.*, 1919, 168, 1111, 1324; 169, 185, 286) that paraffin, naphthene, and aromatic hydrocarbons are very distinctly differentiated by the temperatures above which they are completely miscible with aniline. This temperature for paraffins is high (about 70° C.), for naphthenes about 30°–50° C., while aromatic hydrocarbons are



miscible down to very low temperatures. The addition of aromatic compounds to paraffin and naphthens hydrocarbons lowers this critical solution temperature by an amount very nearly proportional to the concentration of aromatic hydrocarbon present. The curves in Fig. 1 show results of experiments made in this laboratory on the solubility in aniline of a "petrol" freed from aromatic hydrocarbons, and the same mixed with different amounts of benzene, up to 39% by weight. The curves show the temperatures above which mixtures of aniline and the "petrol," represented in composition by the abscissae, become homogeneous. When cooled to temperatures represented by points below the curves separation into two layers occurs. The critical solution temperature in each case is represented by the maximum point; above this point mixtures in all proportions of the respective petrol and aniline are homogeneous.

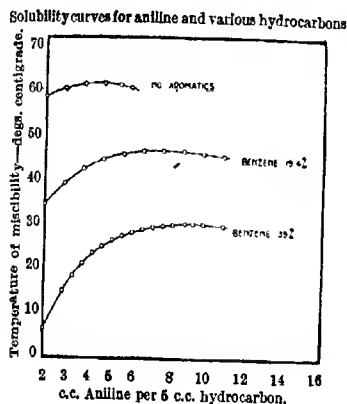


FIG. 1.

The most important feature of these curves is that the proportion of aniline and hydrocarbon at the critical point varies from approximately equal volumes when no aromatic hydrocarbons are present to about 9 volumes of aniline to 5 of "petrol" when the latter contains about 40% of aromatics.

Now, Chavanne and Simon always determine the critical solution temperature, i.e., the temperature represented by the maximum points of the curves in Fig. 1. When this method is used to determine aromatic hydrocarbons it involves following out two such curves, one before and one after sulphonation in every case. This is obviously unpractical, and tends to make the method as laborious as that of the accurate determination of densities. This was recognised at an early stage of the present investigation, and it was decided to measure in every case not the true critical solution temperature, but the temperature of solution of equal volumes of aniline and the hydrocarbon. In practice this comes to the determination of the point of separation into two layers of the "equal" mixture, when it is gradually cooled down from a temperature at which it is homogeneous. This temperature is called by us the "aniline point"; it can be determined with considerable accuracy—certainly to within 0.1° C. with little practice. In the case of mixtures containing very small percentage of aromatic hydrocarbons the aniline point corresponds to the true critical solution temperature within 0.1° C., but when a high percentage of aromatic hydrocarbons is present it may be quite considerably different. In the first case it is obvious from the curves that any possible error in the volumes of aniline and petrol taken causes only a negligible error in the temperatures observed; in the second case it can also be

shown that the error is inconsiderable. When the aromatic content is as high as 39%, a 2% error in volume measurement only makes a difference of 0.3° in the "aniline point," which corresponds in this case to 0.3% of aromatic hydrocarbons, i.e., the error in such an event would be less than 1% of the total aromatic hydrocarbons present. This is an outside estimate of the possible error due to this cause; by always using the same pipettes in measuring out the aniline and the hydrocarbon respectively the volume error is very unlikely to be as much as 2%.

The actual critical solution temperature of aniline and the 39% benzene mixture was found to be rather ill-defined, and it was difficult to get consistent readings to 0.1°, while the "aniline point," i.e., the temperature of separation of equal volumes, was very sharply defined, and could be very accurately determined.

The actual determination of the aniline point is made most conveniently by measuring out from pipettes, 10 c.c. each of aniline and the hydrocarbon into a large test-tube, fitted with a stirrer and a delicate thermometer (reading in 0.2° to 0.1° C.), heating until a clear solution is obtained, then cooling slowly and noting the temperature at which turbidity appears. The mixture is then warmed again very slightly until it is just clear, and the point of turbidity redetermined on cooling. It is necessary to repeat this once or twice in accurate determinations in order to eliminate any error due to possible lag of the thermometer. It is found that a very slight turbidity first appears, and then the mixture suddenly becomes completely cloudy, the thermometer bulb being obscured over a range of about 0.1°. The temperature at which this complete turbidity occurs is taken as the aniline point.

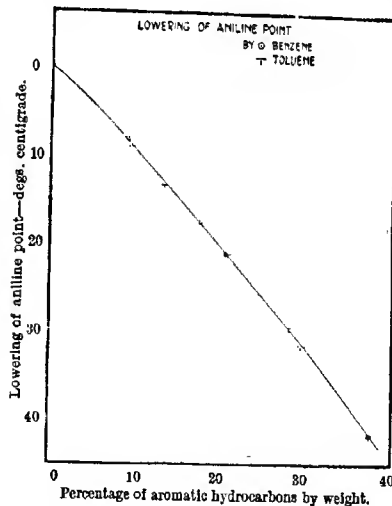


FIG. 2.

The means of heating is a naked flame. When the aniline point is more than 10° above room temperature, the experimental tube is surrounded by a larger tube, so as to leave an air space between the tubes, which ensures that warming and cooling take place gradually. When the aniline point is below room temperature, the tube can be cooled in ice and water, or a freezing mixture. More conveniently the cooling can be accomplished by placing ether in the larger external tube, through which air can be drawn. The apparatus is thus of the greatest simplicity, and from this point of view alone the method has great advantages over those

depending on the measurement of other physical properties such as refractive index or density.

There is further the very considerable advantage, shown during the course of this investigation, that benzene, toluene, and xylene appear to have practically the same effect in lowering the aniline point when added to any mixture of hydrocarbons in the same proportion by weight. Figs. 2 and 3 show the results of special experiments made to find out the effects of the different aromatic hydrocarbons. For these experiments, benzene, toluene, and *o*-, *m*-, and *p*-xylene, were obtained in as pure a state as possible, and the aniline points were determined of special mixtures made up by weight of

(a) Benzene and toluene with a mixture from which all aromatic hydrocarbons had previously been removed by sulphonation (Fig. 2).

(b) The different xylenes with a similar mixture of higher boiling point. In this case also two check mixtures were made up containing benzene and toluene respectively (Fig. 3).

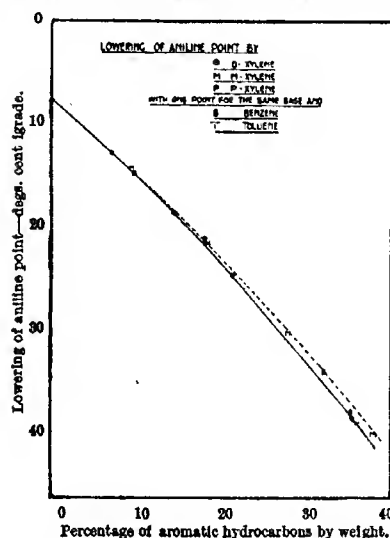


FIG. 3.

Benzene, toluene, and *o*-xylene were found to have identical effects within the errors of experiment, while *m*- and *p*-xylenes lowered the aniline point rather less than the same weight of benzene or toluene. But for all practical purposes it is safe to assume that the same proportions of these hydrocarbons by weight have the same effect in lowering the aniline point, and the following table, from the figures of which Fig. 2 has been constructed, gives the relation between the lowering of the aniline point of a mixture containing no aromatic hydrocarbons and the amount of aromatic hydrocarbon added:—

Percentage of aromatic hydrocarbon in mixture.	Lowering of aniline point.
5% by weight.	4.2° C.
10	8.6
15	12.9
20	17.1
25	21.4
30	25.6
35	29.8
40	34.0

The total aromatic content of any petrol can therefore be determined by this method with considerable accuracy.

The procedure is to determine the aniline point of the petrol, then to shake for  $\frac{1}{2}$  hr. 30–50 c.c. of the petrol with three times its volume of 98%

sulphuric acid, allow to stand, separate the acid, wash the residue with sodium hydroxide solution and water, and dry over calcium chloride. The aniline point of the residue is then determined. The difference between this and the aniline point before sulphonation gives directly the percentage of aromatic hydrocarbons in the mixture. The accuracy has been tested in two cases by making up artificial mixtures by adding known weights of benzene, toluene, and xylenes to a sample from which all aromatic hydrocarbons had previously been removed. The results were:—

Percentage by weight of benzene added	Case I.		Case II.	
	benzene	toluene	benzene	toluene
12.4	12.4	14.6	12.4	14.6
11.3	11.3	11.3	11.3	11.3
Total	33.2	33.2	33.2	33.2
Total found by aniline point method	33.6	33.6	33.6	33.6

If the whole of the added aromatic portion had been *m*-xylene, the error caused by assuming that this hydrocarbon had the same effect as benzene and toluene would be not more than 3% of the total weight added, but naturally this is a circumstance not likely to occur in practice.

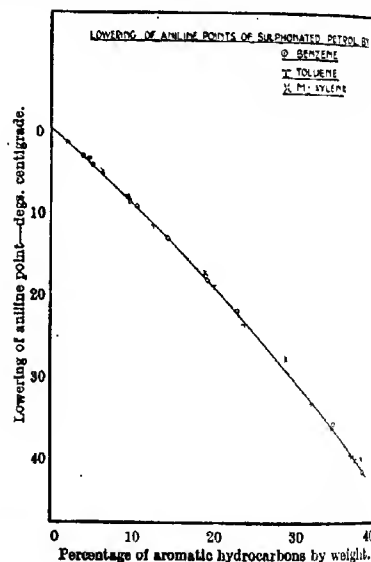


FIG. 4.

The curve is the same as Fig. 2, but the bases used vary widely.

It has been found that when extreme accuracy is not desired the processes of washing and drying the mixture after sulphonation can be omitted. The estimation is then extremely simple, for it is only necessary to pipette off 10 c.c. from the supernatant liquid after sulphonation, and to determine its aniline point immediately. The temperature of turbidity does not differ by more than 0.2° from that of the washed and dried mixture, and the time saved is very considerable.

The above example incidentally gives a proof of the completeness of the removal of aromatic hydrocarbons by shaking with excess of 98% sulphuric acid for  $\frac{1}{2}$  hr. In general our experiments have confirmed Thole's conclusion that the use of acid of this strength prevents action on other hydrocarbons. Very little sulphur dioxide is evolved, and the mixture only gets warm on shaking when there is a large proportion, say 40%, of aromatic hydrocarbons. It is possible that the use of even more dilute acid would be advantageous for analytical purposes, but it has not been thought

worth while to examine this question at present. A special experiment during the sulphonation of a mixture containing 10% of aromatic hydrocarbons showed that nearly all the latter were removed in the first 2½ minutes of shaking.

In another case, when it appeared likely that the non-aromatic portion was attacked by the acid, the residue after the first sulphonation was again shaken with fresh acid. There were undoubtedly signs of a further reaction, but the aniline point after this treatment was found to be unchanged, showing that such action as occurred did not affect the accuracy of the method for determining aromatic hydrocarbons.

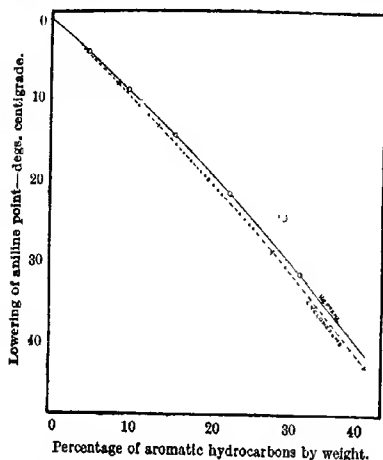


FIG. 5.

Lowering of aniline point of { heptane  
cyclohexane } by benzene.

The next question of practical importance that arises is to what extent the lowering caused by a given weight of aromatic hydrocarbon depends on the nature of the non-aromatic base. Is the lowering, for example, the same when the base is cyclohexane, which has an aniline point of about 30° C., as it is with a pure paraffin such as heptane, which has an aniline point above 70° C.? This would certainly not be expected, and special experiments, made with mixtures of pure specimens of these hydrocarbons with benzene, have shown that it is not the case. The results of these experiments are shown in Fig. 5, the lowering in the case of heptane being less than it is when the non-aromatic base is cyclohexane. The standard curve lies between these two, and the difference between the two curves corresponds to a difference of about 3% of the weight of aromatic hydrocarbons added. But although the nature of the non-aromatic base does affect the degree of lowering, the effect is generally too small to be of practical importance. This is shown more clearly in Fig. 4. In this figure the standard curve of Fig. 2 is reproduced, and the experimental points shown represent results obtained with all the different "natural" petrols so far examined. These petrols range from a Borneo product which has the highest proportion of naphthenes, to American petrol which has the lowest. The aniline points of these fuels after removal of aromatic hydrocarbons vary from 54° C. in the case of former to 64° C. in the case of latter. All the points lie on the same curve within the experimental errors, except those taken with *m*-xylene, to which reference has already been made. It appears, therefore, that in practice the nature of the non-aromatic base has a negligible effect, and

the figures given in Table I. may be taken as standard figures for all natural "petrols."

At the same time, if any doubt exists as to this a check on the accuracy of the determination can easily be obtained in the following way. After the aniline point of the sulphonated residue has been measured, the percentage of aromatic hydrocarbons is calculated approximately from the data given in Table I. (or Curve 2). A solution containing approximately the same weight of benzene, or, better still, two solutions, one containing slightly less, and one slightly more than the calculated result, are made up by adding known weights of benzene to the sulphonated base. The aniline points of these artificial mixtures are determined, and the true aromatic content of the original petrol can be obtained by a very slight interpolation. This procedure is often of value when the non-aromatic base contains a large proportion of low-boiling fractions and has a high aniline point. An actual example will make the method clear. The aniline point of mixture before sulphonation was 38.8° C., and after sulphonation 62.7°. During the latter determination some boiling was observed. A difference of 25.9° C. corresponds to 27.6% of aromatic hydrocarbons from the standard curve. A mixture of the sulphonated product and benzene was then weighed out, containing 28.09% of benzene. The aniline point of this solution was 36.6° C. From the standard curve it is found that when the aromatic content is 28% a difference of 0.2 in the aniline point corresponds to a difference of 0.19 in the aromatic content. Hence this is the correction to be made to ascertain the true percentage of aromatic in the original petrol, which is

$$28.09 - 0.19 = 27.9\%$$

In all the above experiments freshly distilled aniline was used. Commercially "pure" aniline contains small quantities of aminothiophene, the presence of which accelerates oxidation when the aniline is exposed to air and light. In order to ascertain the effect of impure aniline on the aniline point method, moist air was bubbled through aniline for six hours. Experiments made with this product showed a rise of no less than 10° C. in the true "aniline point" of a hydrocarbon base containing no aromatic hydrocarbons. So far as the accuracy of the method for determining aromatic hydrocarbons goes this would be of no consequence if the "lowering" due to addition of aromatic hydrocarbons were the same, but this is not the case.

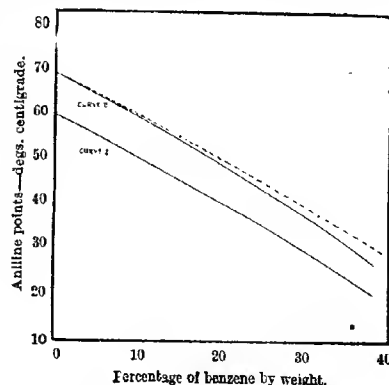


FIG. 6.

Lowering of aniline points by benzene.  
Curve I. Using freshly-distilled aniline.  
Curve II. Using impure aniline.

The lower curve in Fig. 6 represents aniline points taken with freshly distilled aniline; the upper curve shows aniline points of the same mix-

tures with impure aniline. The dotted line has been drawn parallel to the lower curve, and is seen to be of a different slope to the upper continuous curve. Care should therefore be taken to use fairly freshly distilled aniline, but it may be pointed out that all disturbing factors of this kind are eliminated by using the more accurate method outlined above. It would probably be advantageous to use aniline, freed from aminothiophene, or, alternatively, prepared from benzene free from thiophene.

It is important to notice that the purity of the aniline employed seriously affects any analytical methods which depend on the knowledge of absolute aniline points. Chavanne and Simon use the critical solution temperature in aniline to differentiate between naphthene and paraffins etc., and to determine the proportions of such hydrocarbons in petrol. But, owing to the above and other difficulties, an estimation made in this way is only very approximate, although it is often of value to use the method in combination with the density method.

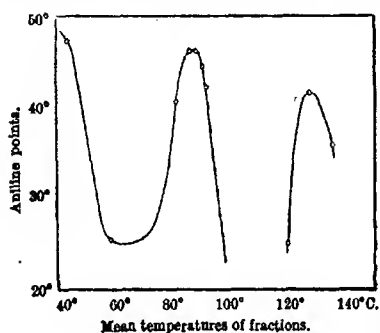


FIG. 7.  
Cutting point curve.

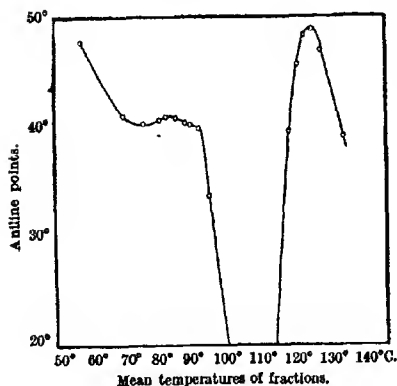


FIG. 8.  
Cutting point curve.

The extension of the aniline point method to the determination of the individual aromatic hydrocarbons involves a separation of the sample by fractionation into fractions containing the benzene, toluene, and xylenes, etc., and separate sulphonation of each of the fractions. In spite of the considerable differences in the boiling points of these aromatic hydrocarbons (benzene 80·4°, toluene 110·8°, xylenes about 139°), it is well known that even when a very efficient fractionating column is used, they distil over wide ranges of temperature. Thole recommends "cutting points" about half

way between the boiling points. He assumes that the fraction distilling over up to 95° contains all the benzene, that between 95° and 122° all the toluene, and that from 122° to 150° all the xylenes. Probably this is not true, but it is reasonable to assume that any benzene distilling over above 95° is balanced by an approximately equal amount of toluene distilling over below 95°. If the densities of the successive fractions are determined it is found that maximum densities occur at about 80°, 110°, and 140°, and minima at about 95° and 122° C. If the aniline points of the successive fractions are determined it is found that maximum points occur near the latter temperatures, indicating that the percentage of aromatic hydrocarbons in the distillates coming over at these temperatures passes through a minimum. It is naturally much quicker to determine aniline points, and if it is considered desirable to ascertain the "cutting points" for any particular fuel as accurately as possible, the petrol is fractionated, and the aniline points of small fractions coming over within small ranges of temperature near 95° etc. are determined. The temperatures at which the aniline points pass through maximum values are taken as the correct cutting points, and a separate fractionation then made.

The following were the results obtained with the two "artificial" petroleums referred to above (Case I. and Case II.). In Case I., in which the proportions of benzene, toluene, and xylene were approximately equal, the cutting points found were 95° and 124° C. (see Fig. 7). Sulphonation of the separate fractions and determination of aniline points as described above gave the following results:—

	Found.	Correct value.
	%	%
Benzene .. ..	12·5	12·4
Toluene .. ..	14·4	14·5
Xylene .. ..	10·6	11·3

In Case II. the proportion of toluene was purposely made high compared with that of benzene and xylene. The aniline points of the successive fractions are shown in Fig. 8, from which it is seen that the maxima occur at 90° and 125° C. Analysis of the fractions obtained by cutting at these temperatures gave:—

	Found.	Correct value.
	%	%
Benzene .. ..	2·4	2·3
Toluene .. ..	16·7	16·5
Xylene .. ..	2·3	3·0

The agreement for benzene and toluene is very good in both cases. The xylene found is a little too low. This may be partly attributed to some uncertainty in the purity of the xylene taken to make up the mixtures, but probably mainly to the fact that the xylene fraction was only one-fifth of the whole in the first case, and one-seventh in the last. It partly consists of the liquid obtained by draining the fractionating column and condenser, and any loss due to incomplete drainage tends to make the xylene value rather too low.

The errors that would have been caused by taking the normal cutting points of 95° and 122°, instead of 90° and 125° may be estimated in the following way.

Between 90° and 95°, 40 c.c. distilled over, containing about 10% of aromatic hydrocarbons. The total amount of the product distilled was 500 c.c., so that this represented

$$\frac{1}{10} \times \frac{40}{500} = 0·8\%$$

of aromatics in the total. If this had been included in the benzene fraction it would have raised the benzene value to 2·4+0·8=3·2%, whereas the actual benzene content was 2·3%.

On the other hand only 8 c.c. distilled between 122° and 125°, and the distillate contained 8%

aromatics. Had this been included in the xylene fraction, it would have raised the percentage of xylene by  $8 \times 8 + 500 = 0.13$ , and the final values would have been benzene 3.2%, toluene 15.8%, xylene 2.4%. Hence in extreme cases, when the aromatic content consists mainly of one hydrocarbon, the error due to assuming the correct cutting points to be 95° and 122°, without making a special determination, may be fairly considerable, but for all ordinary cases these cutting points are sufficiently accurate.

In conclusion, to show the degree of agreement found in actual analyses between the total aromatic content, and the sum of the individual hydrocarbons, the experimental figures for a number of products examined in this laboratory are quoted:—

Fuel.	Benzene. %	Toluene. %	Xylene and higher aromatics. %	Total. %	Total found independently. %
(1)	8.1	16.3	16.7	39.1	38.6
(2)	7.2	17.5	10.3	35.0	35.2
(3)	6.8	8.0	1.3	14.9	14.6
(4)	4.9	4.1	0.0	9.9	10.0
(5)	0.7	3.8	3.8	8.3	8.6
(6)	2.7	4.4	1.8	8.9	8.4
(7)	12.7	4.9	2.2	9.8	10.1
(8)	1.0	1.8	1.4	4.2	4.4

It should be remembered that as outlined above this method is only applicable to the estimation of aromatic hydrocarbons in mixtures which contain negligible quantities of unsaturated hydrocarbons. The presence of the latter introduces a serious complication, which will be dealt with in a later paper. The method has now been in use for some time with satisfactory results in the laboratories of the Asiatic Petroleum Company, to the directors of which we are indebted for permission to publish the above results.

Organic Chemical Laboratory, Oxford.

#### SOLUTIONS OF ACETYLENE IN ACETONE AT HIGH PRESSURES.

BY RAYMOND E. BUTLER, B.Sc., A.I.C.

In the course of experiments in connexion with the solution of acetylene in acetone at high pressures, it became necessary to obtain a knowledge of the rate of expansion of a given volume of acetone when saturated with acetylene at pressures in the neighbourhood of 10 atmospheres.

An examination of the literature on the subject revealed a remarkable dearth of information.

Claude and Hess (Comptes rend., 124, 626) showed that the increase of solubility of the gas in acetone is nearly proportional to the pressure. This is the basis upon which the dissolved acetylene industry was founded. They also showed that when saturated at a pressure of 1 atm. the acetone increased in volume by about 4%.

Siller ("Versuche über gelöstes Acetylen," 1913) found that the expansion factor was 4.7% up to a pressure of 75 lb. per sq. in.; no investigation was made at higher pressures.

It was therefore decided to investigate the nature of the solution at higher pressures, and in view of the danger attending the compression of the acetylene at pressures greater than 2 atm. specially designed apparatus was necessary.

##### Method of storage of gas.

Since gas containing acetone vapour, as delivered from cylinders of dissolved acetylene, was useless for our purpose, the gas was stored in a cylinder of 2 cub. ft. capacity filled with beech charcoal, of Belgian manufacture, of absolute sp. gr. 1.41.

It was desired to obtain a porosity of about 80% in the gas storage cylinder, the water capacity of

which was 124 lb. 10 oz. weight of water. It was therefore necessary to fill the cylinder with 35 lb. 3 oz. of charcoal. The cylinder so prepared was completely exhausted of air and occluded gases by subjecting it to a hot vacuum process at 100° C. for 24 hours. It was then cooled, and when cool filled with acetylene by means of a compressor.

The acetylene used was obtained from calcium carbide in a carbide-to-water generator, from which it passed through a hydraulic main, purifiers containing chromic acid mixture, and dust traps, previous to entering the compressor.

The gas was allowed to enter the cylinder from the compressor slowly in order to avoid local overheating, with consequent danger of explosion. Acetylene compressed at a pressure greater than 2 atm. is liable to decompose with violence unless absorbed in a porous mass the interstices of which have an average diameter of less than 0.5 mm. (Le Chatelier).

On disconnecting from the compressor, and cooling, the cylinder was found to contain gas at a pressure of 175 lb. per sq. in.

##### Absorption apparatus.

The apparatus used for the absorption experiments is illustrated in Fig. 1. It consists of a constant temperature bath, F, through which a

steady stream of water flowed from a thermally-regulated constant-temperature cistern; an inner glass pressure tube, B (34 in. in length, approx. diam.  $\frac{1}{2}$  in.), held by means of tie-rods, H, between metal plates, G, G, and making a gas-tight joint with each plate by means of a fibre seating (see Fig. 2); an outer steel safety tube, C, surrounding B, and having a narrow slit running from end to end, through which the level of the acetone could be observed, and its height obtained accurately on a metal scale (not shown).

Since it is impossible to obtain acetylene of 100% purity by any ordinary method, the gases other than acetylene (which are but slightly soluble in acetone) were allowed to escape by means of the gas outlet valve, E, which served as the fine-adjustment valve for each high pressure reading.

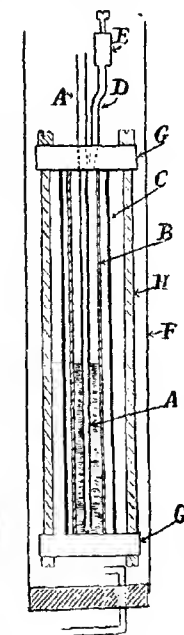


FIG. 1.

The compressed gas from the storage cylinder was admitted by way of a standard pressure gauge into A, whence it bubbled up through 20 c.c. of acetone in B, collecting in the space above the acetone and establishing the necessary pressure on the surface. When the gas passed through without dissolving (this was readily indicated by the bubbles) the excess of gas was allowed to escape slowly through E.

The pressures were then finally adjusted by means of E so that the state of balanced pressures was obtained, and the acetone rose inside A. Each reading was taken under the same conditions, the

height being read when the level of the acetone inside A was identical with that of the main bulk of acetone surrounding A. Great care was necessary in the final adjustments made by the valve, E, as it was found that any slight irregularity in the glass below the surface of the acetone had a tendency to form a centre from which bubbles of

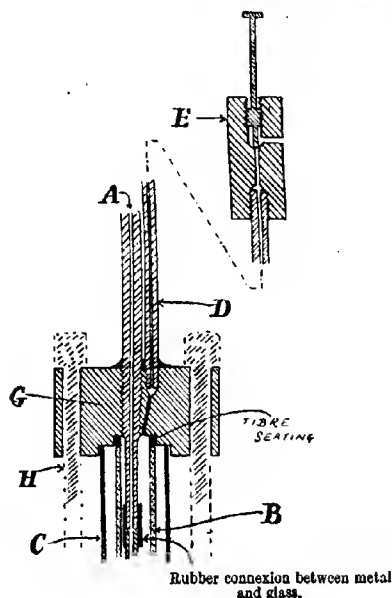


FIG. 2.

dissolved gas attempted to escape from solution.

The acetone used had b.p.  $56.5^{\circ}\text{C}$ ; sp. gr. 0.794 at  $15^{\circ}\text{C}$ , coefficient of acetylene absorption, 23.8 volumes at  $15^{\circ}\text{C}$ .

The readings were taken at  $15.8^{\circ}\text{C}$ . It was found by experiment that 1 cm. height of acetone in inner tube B = 0.472 c.c. The following results were obtained:—

Absolute pressure, lb. per sq. in.	Scale reading, in cm.	Increase in length, in cm.	Expansion in c.c.	Increase in volume, %
0	40.8	—	—	—
15	42.5	1.7	0.80	4.0
30	44.6	3.8	1.80	9.0
45	46.4	5.6	2.64	13.2
60	48.4	7.6	3.60	18.0
75	50.0	9.2	4.34	21.7
90	52.7	11.9	5.62	28.1
125	56.4	15.6	7.36	35.8
145	58.8	18.0	8.50	42.5

The above results when plotted on a curve, with pressures as abscissae, and expansions as ordinates, show that up to a pressure of 10 atm. (absolute) the expansion follows a straight-line law, the percentage expansion per atmosphere increase in pressure being 4.4%.

This figure is important, in view of the Home Office order permitting the filling of dissolved acetylene cylinders to a pressure of 15 atm. It is obvious that under these conditions the acetone in the cylinder may expand by as much as 64% of its original volume.

In a cylinder containing 40% of its volume of acetone, the final volume of the acetone, calculated from this factor, is 65.6% of the volume of the cylinder. In addition, an ordinary dissolved acetylene cylinder, filled with porous material having a porosity of 40%, has a total volume of solid matter within it of 20% of its volume. The

total free space actually existing in the charged cylinder is thus only 14.4%.

This figure must be still further reduced should the acetone expand, due to any pronounced increase of temperature of the cylinder and contents. There is no evidence that any compound is formed between the solvent and the solute. Up to a pressure of 10 atm. absolute the phenomenon appears to be entirely physical in nature.

These results were obtained in the course of a series of experiments carried out in the laboratories of the Dissolved Acetylene Company, London, S.W. 8.

#### NOTE ON THE RATE OF REACTION OF PICRIC ACID WITH NITRATING ACID.

BY D. L. HAMMICK.

During the course of some experiments on the yields of picric acid obtainable by carrying out the nitration of phenol at higher temperatures than are usual, it became necessary to ascertain the rate of reaction between picric acid and the nitrating acid. The following experiments were therefore carried out.

A nitrating acid was made up of equal parts by weight of water, nitric acid (68%), and sulphuric acid (sp. gr. 1.345). Pure, recrystallised picric acid was dissolved in excess of this mixture and heated in a 250-c.c. Kjeldahl flask in a constant temperature oil bath. Loss of water and nitric acid by evaporation was prevented by the insertion of a close-fitting glass condenser into the neck of the flask. Portions of the picric acid solution were removed from time to time, diluted, extracted with ether, and the extract evaporated and dried at  $100^{\circ}\text{C}$ . The residual picric acid was then estimated with titanous chloride by Knecht and Hibbert's method.

The results obtained are given below. The initial concentration,  $a$ , of the picric acid is in grams per 100 c.c. of solution;  $a-x$  is grams picric acid in 100 c.c. of solution after time  $T$  (in days).  $K$  is the unimolecular velocity constant in the equation.

$$K = \frac{2.3}{T} \cdot \log \frac{a}{a-x}$$

Temperature.	$115^{\circ}\pm 1^{\circ}\text{C}$ .		$106^{\circ}\pm 1^{\circ}\text{C}$ .		$96^{\circ}\pm 1^{\circ}\text{C}$ .	
$a$ , g. per 100 c.c.	0.309		1.193		1.813	
T	$a-x$	K	$a-x$	K	$a-x$	K
2	0.490	0.25	0.920	[0.130]	1.657	[0.045]
3	0.368	0.25	—	—	1.844	0.033
4	0.291	0.26	0.838	0.089	1.600	0.031
5	0.248	0.24	—	—	1.542	0.032
6	0.202	0.24	0.623	[0.110]	—	—
7	0.164	0.23	—	—	1.469	0.030
8	0.120	0.23	0.608	0.085	—	—
9	—	—	0.570	0.083	1.381	0.030
10	—	—	0.504	0.086	—	—
11	—	—	—	—	1.299	0.030
12	—	—	0.417	0.088	—	—
13	0.032	0.25	—	—	—	—
Mean .. ..	0.245		0.086		0.031	
Period of half-change ..	2.76 days		8.1 days		22.3 days	

It is apparent from the above results that even at comparatively high temperatures the rate of destruction of picric acid by nitrating acids is too slow to affect the yields obtained by the nitration of phenol.

Oriel College, Oxford.

#### ERRATUM.

In the discussion on the paper by S. J. Peachey and A. Skipsey (J., Jan. 15, 1921, p. 5 r) for "Mr. Bailey" (col. 2, line 9 from bottom) read "Mr. F. Bayley."



## Bristol and S. Wales Section.

Meeting held at Bristol on Dec. 2, 1920.

MR. F. WALLS IN THE CHAIR.

## THE HYDROLYTIC ALKALINITY OF PURE AND COMMERCIAL SOAPS.

BY F. C. BREDLE AND T. R. BOLAM.

In two earlier communications from the Bristol laboratory,\* which constitute the only quantitative measurements of hydrolysis in soap solutions, it was shown that the hydrolysis of soaps was unexpectedly slight.† The two independent methods involving E.M.F. measurements and catalysis prove that the hydrolytic alkalinity of soap solutions is negligible for most purposes since the concentration of hydroxyl ion is only about  $N/1000$ .

The object of the present paper was to obtain further data for pure salts of the fatty acids and to investigate soaps made with single typical oils as well as commercial soaps.

## Experimental method.

Of the two available methods for the determination of hydroxyl ions in such solutions, the E.M.F. method suffers in the investigation of commercial soap from the serious drawback that the potential of the hydrogen electrode will probably be affected by the presence of unsaturated compounds. The method of the catalysis of nitrosotriacetone-amine‡ to give phorone was therefore adopted. The reaction is unimolecular, and the velocity is proportional to the concentration of hydroxyl ion. The volume method had been found to be accurate to within about 2 or 3% at the ordinary temperature.

The results are calculated according to the system proposed by McBain (Trans. Faraday Soc., 1917, 13, 1), which consists in always setting the rate-constant equal to unity and in choosing the unit of time accordingly.

The formula used here is consequently

$$T = 2.303 \log. A/(A-x)$$

instead of the hitherto customary

$$k = 2.303/t \log. A/(A-x)$$

where  $k$  is the rate-constant. The disadvantages of the system hitherto employed, as compared with that proposed, are illustrated by the rate of reaction in the presence of  $0.000656N$   $OH'$  at  $90.0^\circ C$ . The statement that "the rate-constant is 0.112" is meaningless until the further information is given that "time has been measured in minutes." Under the new system this becomes—"the time unit is 8.9 minutes"—a statement which is much more readily visualised, is self-contained, and does away with small decimals. It further means, in the case of unimolecular reactions such as the present, that the reaction requires 8.9 minutes to proceed to the extent of 63.2% or nearly two-thirds of completion.

The chief difficulty to be avoided in this method is the sorption of the amine by the soap in concentrated solution; so that dilute solutions containing not more than 1 g. in 100 c.c. of water had to be employed. It was soon found too that this influence persisted much more strongly at lower temperatures, and hence the temperature of  $90^\circ C$  was adopted for the present experiments.

\* McBain and Martin, *Chem. Soc. Trans.*, 1914, 105, 968, and McBain and Bolam, *ibid.*, 1918, 118, 825.  
† Cf. Lewkowitch, *J.*, 1907, 590, who considered that hydrolysis would prove to be so great that complete saponification could not be obtained except in presence of a large excess of alkali.  
‡ Francis and collaborators, *ibid.*, 1912, 101, 2968; 1913 105, 1722; McBain and Bolam, *loc. cit.*

The values of the time units for definite concentrations of hydroxyl ion were measured by observing the rate in solutions of sodium hydroxide. These solutions were made up from sodium and boiled-out conductivity water. The reaction was carried out in a silver flask containing a small glass tube, which could be dropped into the solution in the flask when it was desired that the reaction should commence. In some cases this tube contained the soap solution, in others the solution of nitrosotriacetone-amine; 0.1300 g. of that substance was employed in each case.

The concentrations of the soap solutions are expressed in weight normality; that is, gram-equivalents of the soap per 1000 g. of water in the case of the pure soaps such as the oleate. The commercial soaps are expressed in "percentages"; that is, the number of grams of commercial soap contained in 100 c.c. of solution at room temperature. The values given in this column refer in the cases of sodium oleate and abietate to grams of anhydrous fatty acid in 100 c.c. of solution. The concentration of hydroxyl ion, on the other hand, is invariably expressed in volume normality at  $90^\circ C$ .

It is essential to flush out the vessels used with air free from carbon dioxide. Although with the soaps of the pure saturated fatty acids constant results were obtained for the time units and reaction constant throughout the whole course of any one reaction, nevertheless in most of the present cases which involve unsaturated fatty acids it was found that there is a tendency of the velocity of the reaction to fall below the value required for the unimolecular formula after the reaction has proceeded part way. This is possibly due to slight oxidation, and hence in all such cases the constant initial part of the reaction was taken as the basis of calculation of the hydrolysis-alkalinity given in the tables. A blank test showed that a solution of sodium oleate did not absorb a volume of air sufficient to affect the volume measurements.

In the first series of experiments carried out by one of us (T. R. B.), the value for the product  $CT$ —that is, concentration of  $OH'$   $\times$  value of time units at  $90^\circ$ —was found to be 0.00561, and this value was used for calculating with olive oil, coconut, toilet and cold process soap. In the remaining experiments (by F. C. B.) the value  $CT = 0.00583$  was used.

In all cases here recorded experiments were carried out at least in duplicate, and about ten readings were made in each experiment. The experimental results are summarised in Tables I. and II. The many interesting points arising are discussed under the separate headings below.

## Pure sodium oleate.

Since all the data for pure soaps so far published refer to solutions prepared from pure saturated fatty acids, it is desirable to supplement them with data for an unsaturated fatty acid for comparison, particularly as this is an important constituent of some of the soaps studied here.

Oleic acid "Kahlbaum" was employed, and the solutions were prepared by Miss Laing, as in previous communications, using a quantity of alkali equivalent to the titration value in aqueous alcohol determined experimentally.

Examining the results of the tables, it is seen that the hydroxyl concentration (hydrolysis-alkali) passes through an absolute maximum in  $N/20$  sodium oleate solution. The actual percentage hydrolysis is negligible at high concentration, but increases rapidly in very dilute solution.

Comparison with the previously communicated results of McBain, Bolam, and Martin shows that the hydrolysis-alkalinity of the oleate amounts to almost exactly as much as in the sodium and potassium palmitate.

TABLE I.  
Rate of catalysis of soap solutions at 90° C.

Soap studied.	Conc. soap.	Time units in minutes.			Conc. OH <sup>1</sup> .	Hydrolysis, %
		max.	min.	mean.		
Sodium oleate .. ..	0.1000 N = 2.7%	7.60	7.22	7.38	0.00079	0.81
" .. ..	0.0500 N = 1.4%	8.14	5.77	5.96	0.00098	1.09
" .. ..	0.0250 N = 0.6%	8.33	7.65	7.94	0.00074	3.7
" .. ..	0.0100 N = 0.3%	9.23	8.29	8.88	0.00066	6.6
" .. ..	0.0050 N = 0.04%	10.91	10.31	10.60	0.00055	28.1
Sodium abietate .. ..	0.0243 N = 1.0%	6.44	5.41	5.97	0.00098	4.0
" .. ..	0.0121 N = 0.5%	8.71	8.21	8.52	0.00069	5.7
Olive oil .. ..	1.0%	7.69	7.55	7.64	0.00073	—
" .. ..	0.5%	9.90	8.70	9.30	0.00060	—
" .. ..	0.1%	32.35	30.69	31.47	0.00018	—
Coconut oil .. ..	1.0%	18.08	18.02	18.05	0.00031	—
" .. ..	0.5%	21.41	24.51	26.46	0.00021	—
" .. ..	0.1%	—	—	122.7	0.00005	—
Milling base .. ..	1.0%	7.46	7.30	7.38	0.00076	—
" .. ..	0.5%	9.89	8.85	9.33	0.00060	—
" .. ..	0.1%	23.35	23.26	23.31	0.00024	—
Cold process .. ..	1.0%	5.25	5.13	5.19	0.00108	—
" .. ..	0.5%	7.19	6.76	6.98	0.00080	—
" .. ..	0.1%	18.79	18.59	18.69	0.00030	—
" .. ..	0.5%	7.76	6.90	7.19	0.00081	—
Soluble washer .. ..	0.5%	7.66	7.38	7.59	0.00077	—
Tallow rosin .. ..	0.5%	7.17	7.57	7.34	0.00079	—
Coal tar .. ..	0.5%	7.24	7.06	7.14	0.00082	—
Shaving .. ..	0.5%	6.48	5.92	6.24	0.00094	—

\* An alkalinity of 0.001 N OH<sup>1</sup> corresponds to a hydrolysis of about 5% in the case of the solutions containing 1 g. of soap in 100 c.c. of water.

TABLE II.  
Hydrolysis-alkalinity (OH<sup>1</sup>) of soap solutions at 90° C.

Soap.	1%.	0.5%.	0.1%
Coconut oil .. ..	0.00031	0.00021	0.00005
Olive oil .. ..	0.00073	0.00060	0.00018
Toilet soap .. ..	0.00076	0.00060	0.00024
Cold process .. ..	0.00108	0.00080	0.00030
Washer .. ..	—	0.00077	—
Tallow rosin .. ..	—	0.00079	—
Coal tar .. ..	—	0.00082	—
Shaving .. ..	—	0.00094	—
Sodium oleate .. ..	0.00074	0.00066	0.00055
Sodium abietate .. ..	0.00098	0.00069	—
Sodium palmitate .. ..	0.00091	—	—
do. (K.M.F. method)	0.00190	0.00066	—

In the case of all these soap solutions, it is probable that the maximum concentration of hydroxyl ions observed in about  $N/20$  solution is quite real. Whilst in more dilute solution the percentage hydrolysis rapidly rises, this is counterbalanced by the diminishing concentration of the soap itself. With increasing concentration, on the other hand, the concentration of hydroxyl ion drops off owing to the gradual disappearance of the simple fatty ion and its replacement by colloidal ionic micelle, for it is the simple fatty ion only that is hydrolysed and the low values for the hydrolysis-alkalinity found with the catalytic method at high concentrations of soaps are only partially ascribable to the sorption of amine by soap. The attainment of the maximum hydroxyl ion concentration in the comparatively dilute ( $N/20$ ) solution of sodium oleate is characteristic of the very colloidal nature of the oleates, since the oleate is more colloidal than the palmitate (maximum  $N/10$ ), and very much more so than the abietate ( $>N/10$ ).

#### Sodium abietate (rosinate).

For this constituent of washer and household soaps there were likewise no available data. The material employed was a specimen prepared by Miss K. M. Gibbons from best W.W. rosin. The rosin was boiled in anhydrous alcoholic solution of sodium ethoxide (free from carbon dioxide) for many hours in order to ensure complete saponification, and very carefully adjusted to give an exactly neutral solution when samples were tested with phenolphthalein in aqueous alcohol free from carbon dioxide. There-

upon the sodium salt was completely dried and analysed by Miss Laing.

The data in the tables show that the "abietic" acid is about as strong as palmitic and oleic acids, but that its salts being less colloidal, are more hydrolysed in stronger solutions. This agrees with other evidence which would place sodium rosinate amongst the lowest soaps.

#### Coconut oil and olive oil soaps.

Coconut oil is known to be a constituent of certain free-lathering soaps, some of which are said to have an action upon the skin, and olive oil soap is especially recommended for sensitive skins.

Thus Simmons\* states: "The soap made from coconut oil has a rather irritating effect on sensitive skins, consequently the proportion of coconut oil in fate to be saponified for toilet soap should never exceed 25%," and again, "olive oil forms a very mild neutral soap especially adapted for sensitive skins . . . not a very good detergent."

It appeared of interest to inquire whether the irritant action of coconut oil soap was due to great hydrolytic-alkalinity or to some other cause, such as the presence of an active constituent or perhaps a specific action of the laurates themselves, which form the chief constituent of the oil.

Two special soaps were prepared from coconut oil and sulphur olive oil, respectively, through the kindness of Messrs. Christopher Thomas and Bros., Ltd., in lots of about  $\frac{1}{2}$  ton apiece. These were boiled in the ordinary way and "fitted" with the utmost care to produce a neutral settled soap. This was important, as the papers referred to have shown that any free alkali will partly remain present as such in the solution, and the hydrolytic-alkalinity would therefore be increased to that extent. Analysis of the olive oil soap showed only 0.003% of free alkali. These soaps were diluted with boiled-out conductivity water to form the solutions studied.

The tables show at a glance that, contrary to expectation, the coconut oil soap solution contained even less OH<sup>1</sup> than the olive oil soap. Free alkali could only account for 3.5% of the total hydrolytic alkalinity.

These data dispose of the presumption that soap solution with coconut oil as a constituent are more alkaline than those containing olive oil. Hence

\* "Soap," W. H. Simmons, pp. 9, 10. (Pitman's "Commodity of Commerce" Series.)

irritation of the skin generally stated to be produced by a commercial free-lathering soap in dilute solution must be ascribed to something other than the actual alkalinity of the solution.

Evidence confirmatory of these views is to be found in the results for a soluble washer household soap as compared with a mere old-fashioned type of tallow-rosin household soap. The hydrolytic-alkalinity in both of these is identical (0.00077N and 0.00079N), and is nearly four times greater than that of a soap made from coconut oil alone.

Indeed, it would appear that the hydrolytic-alkalinity of soap solutions is essentially that produced by the most hydrolysable constituent, the others having but little effect.

#### Toilet soaps.

The tables include measurements carried out on a milling base for toilet soap containing 0.078% of free alkali by analysis. It will be noticed that the alkalinity of this soap is very slightly more than that of the pure olive oil soap.

McBain and Martin have shown that the degree of alkalinity of soap solutions rises rapidly as the homologous series of fatty acids is ascended, and, of course, as indicated above, the most highly hydrolysed constituent of the solution will be the chief controlling factor in the determination of the hydroxyl ion. This would ascribe the high degree of hydrolysis to the important amounts of higher saturated fatty acid present, although the alkalinity falls appreciably short of that of a pure sodium palmitate solution, being in this case about 75% of the value obtained for the latter. This bears out the results obtained by McBain and Martin with toilet soaps in much more concentrated solution. Household soaps, on the other hand, they found to be less alkaline than the toilet soaps, but the values obtained in this case were open to serious doubt on account of the effect of unsaturated constituents on the hydrogen electrode.

In dilute solutions the household soaps are not very much more alkaline than the best toilet soap, and, if anything, rather less so than the coal tar soap, which is sometimes recommended by the medical profession for sensitive skins. The most alkaline soap examined is an imported shaving soap, probably because it contains so much stearate.

#### Cold process toilet soap.

Simmons also states (*loc. cit.*, page 37) that "since fats and oils can never be completely saponified without an excess of alkali, and it is obviously impossible to add such an excess to a cold process soap without getting a strongly alkaline soap . . . cold process soaps always contain small amounts of free acid and uncombined alkali . . . and uncombined alkali has, of course, an irritating action on the skin."

Consideration of the low temperature-coefficient of heterogeneous reactions would make this statement appear very doubtful, and the evidence of the measurements in the tables show that saponification is complete. The specimens used were samples of commercial soap of English manufacture, and the measurements were made as soon as possible after making up the solutions. The alkalinity of the solutions is so like that of the other soaps that it is probably all due to hydrolysis.

#### Sodium carbonate.

Simmons (*loc. cit.*, p. 25) states that "sodium carbonate has an irritating effect on sensitive skins, and consequently must only be present to a very small extent in a toilet soap, usually not exceeding 0.15%. The experiment gave a concentration of hydroxyl ion in 0.15% sodium carbonate solution at 25°, amounting to 0.0010 N. This is very interesting, for although alkalinity rises rapidly with rise of temperature, very hot water cannot be employed in washing. Thus a maximum alkalinity recognised

as permissible is about 0.001—0.002N OH', which is exactly equal to the maximum alkalinity hitherto recorded for a commercial soap, namely in a well-known imported shaving soap measured at 90° in rather high concentration by McBain and Martin (*loc. cit.*; see also Tables I. and II. for the dilute solution), and which is at least twice as much as obtains for any of the concentrations of soap solutions included in Tables I. and II.

#### Summary.

1. The free alkali contained in any well-made soap, intended to be neutral, accounts for only a fraction of the alkalinity (OH') which is observed in dilute solutions of soap and which is due to hydrolysis.

2. The hydrolysis-alkalinity of dilute soap solutions rarely amounts to N/1000; that of concentrated solutions not more than N/500. The latter alkalinity is that which corresponds to about 0.15% of sodium carbonate, larger quantities of which should not be found in toilet soaps.

3. Sodium oleate and the sodium salt of rosin are about as much hydrolysed as the palmitate, but in stronger solutions the oleate is less and the rosinates more hydrolysed in correspondence with their respective degrees of colloidal properties.

4. Soap prepared from pure coconut oil has an exceptionally low hydrolytic-alkalinity. Neutral soap from pure olive oil exhibits a hydrolytic-alkalinity similar to good toilet soap and slightly exceeded by household soaps and some other toilet soaps.

5. The hydrolytic-alkalinity of a soap solution appears to be chiefly conditioned by the most hydrolysable constituent present. Thus shaving soap exhibits a high degree of hydrolytic-alkalinity. Similarly, the hydrolytic alkalinity arising from coconut oil soaps does not account for any irritating action on the skin. Such action must be ascribed to some other constituent present, such as, possibly, a specific action of the laurates contained in it. Soaps consisting largely of the more readily sorbed soaps of the higher fatty acids should not exhibit irritating action upon sensitive skin, as in these mixtures sorption of such constituents as the laurates by the skin will be largely obviated.

6. A free-lathering soluble washer household soap exhibits about the same hydrolytic alkalinity as a tallow rosin (0.0008N OH').

7. A well-made cold-process soap does not exhibit hydrolytic-alkalinity appreciably greater than could be expected from any toilet soap made from the same materials. In other words, the OH' concentration is too small to have an effect even upon sensitive skin.

In conclusion, we desire to thank Prof. McBain for the interest he has shown in our work, which was undertaken at his suggestion, and the Colston Society of the University of Bristol and the Chemical Society of London for grants in aid of the investigation.

## London Section.

Meeting held at Burlington House, on January 3, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

### THE ANALYSIS OF LIQUID AND GASEOUS MIXTURES OF ETHER, ALCOHOL, AND WATER.

BY IRVINE MASSON AND T. LAWSON McEWAN.

Despite the widespread applications of ether-alcohol as a solvent on the large scale, especially in the nitrocellulose industries, the published methods of analysis of these mixtures are either tedious or

unreliable, and in any case approximate only; and for the analysis of mixtures of the vapours diluted with air, which should serve to control processes for the recovery of evaporated solvent, satisfactory methods appear to have been entirely lacking (cf. Mallinckrodt and Alt, *J. Ind. Eng. Chem.*, 1916, 8, 807; Wolff, *Chem.-Zeit.*, 1910, 34, 1193; Kochmann and Strecker, *Biochem. Zeits.*, 1912, 43, 410). The methods now described were therefore devised and tested, and were adopted at Gretna and other cordite factories during the war.

Fleischer and Frank (*Chem.-Zeit.*, 1907, 31, 665) described an approximate method in which the aqueous ether-alcohol sample was shaken with water and paraffin ("Benzin"), the increase in volume of the oil being taken as the volume of ether present. From this and from the density of the sample the apparent density of the alcohol-water portion was algebraically calculated, and the strength of this found from ordinary alcohol tables. It was thus assumed that all the ether was extracted by the oil, and, further, that no change in volume takes place on mixing ether with aqueous alcohol. As the sequel shows, neither assumption is correct, and the results deviate from the truth by about 10% for the ether and by much more for the alcohol.

By using extraction with paraffin and water, however, if due allowance be made for the partition of ether and alcohol between the two layers, the ether content of a sample can be found within less than 1% of the truth; and if this result and the density of the sample are compared with actual experimental data for the densities of synthetic ternary mixtures, the alcohol content may be found with an accuracy of 1%.

In the case of vapours similar principles apply to the ether estimation, the vapours being first absorbed in sulphuric acid.

#### A.—Liquid mixtures.

Paraffin, water, ether, and alcohol together form two layers, of which the upper contains all the paraffin, most of the ether, and a part of the alcohol. If sufficient water be present to reduce the concentration of alcohol in the aqueous layer below about 25%, little alcohol can pass into the upper layer with the ether; similarly, excess of paraffin improves the extraction of ether into the upper layer. These conditions are met if 1 vol. of the sample is shaken with 2 vols. of paraffin and 2 of water. In these circumstances the volume of the paraffin increases by an amount which may be between 80% and 105% of the volume of ether actually present, according to the kind of oil used and, to a slight extent, on the ether content of the sample tested.

The light paraffins give the highest apparent efficiency of extraction; heavier varieties, such as centrifugal oils and paraffin "B.P." give emulsions or are too viscous. Two qualities are here specified. For ordinary purposes by much the best is petroleum ether, of sp. gr. 0.68 at 20°/4°, b.p. 40°–75° C. For special cases, to be referred to later, a mobile paraffin of sp. gr. 0.75 at 20°/4°, b.p. 120°–140° C., is used; lamp oil, even after purification, is an unsatisfactory substitute. A convenient source of the oil is the xylene fraction of Borneo oil, as obtained from the Asiatic Petroleum Company; this oil is treated at 100° with oleum to remove aromatic and unsaturated hydrocarbons, and is shaken successively with alkali, water, anhydrous calcium chloride, and mercury, being finally filtered. Other sources are doubtless available.

Emulsification is avoided by the use of water containing a little acid (2% H<sub>2</sub>SO<sub>4</sub>) for the extraction.

Finally, owing to the high coefficients of expansion of ether and of petroleum ether, due care must be taken in analysis to counteract the rise of temperature which occurs on mixing alcohol with

water. The actual distribution of the components seems to be little affected by this temperature-change, so that where weighing is adopted no such precaution is necessary.

#### (a) Estimation of ether.

(i.) *Large samples.*—A separating-funnel containing 200 c.c.±1 of petroleum ether as already specified is weighed to 0.1 gram. 100 c.c.±0.5 of the sample is added and the weight again taken. 200 c.c.±1 of water containing 2% H<sub>2</sub>SO<sub>4</sub> is added, without weighing, and the corked vessel is vigorously shaken, after which it is allowed to stand for a few minutes with occasional rotation. The aqueous layer is now run off so that a few c.c. remains, and the corked vessel allowed to stand for ten minutes, adherent drops of moisture being released by intermittent swirling. The aqueous layer is now finally run off and the vessel and contents weighed. The total time required for the analysis is 20 minutes.

The gain in weight of the oil is about 90% of the true weight of ether present; the true value is found from a curve which should be drawn from the determinations with synthetic mixtures recorded in Table I. The accuracy of the result is within 1%, excepting where much water is present in the sample, when the value read from the curve is low by 1% (see Table I.).

TABLE I.

*Ether extraction by petroleum ether.—Weight method.*

(Ether-alcohol-water, 100 c.c.; p.e., 200 c.c.; 2% H<sub>2</sub>SO<sub>4</sub>, 200 c.c.)

Mixtures with 94% spirit.			Mixtures with 75% spirit.		
Ether, g.	Gain in p.e.	Ratio.	Ether, g.	Gain in p.e.	Ratio.
0	0.4	—	16.9	14.9*	0.88
5.2	5.0	0.96	32.6	29.3	0.90
16.3	14.6	0.90	59.8	54.3	0.91
23.4	21.3	0.91	Mixtures with 60% spirit.		
30.9	27.5	0.89			
36.7	33.6	0.92	17.8	15.6	0.88
45.7	41.7	0.92	33.9	30.7	0.92
53.3	49.3	0.93			
58.1	53.1	0.92			
71.2	65.1	0.92			

(ii.) *Small samples.*—Where only 20–30 c.c. of liquid is available the following method is accurate. A burette of 100 c.c., graduated in tenths, has bound to it a thermometer, with its bulb kept closely in contact with the burette at about the 30 c.c. mark by means of rubber rings. A jacket of cotton wool is wrapped around the bulb and burette at this point. Mercury is put in up to the lowest mark; 40 c.c.±0.1 of water containing 2% H<sub>2</sub>SO<sub>4</sub> is added, and 40 c.c.±0.1 of petroleum ether. The burette is corked, and after 5 minutes' settling the exact volume of the upper layer is read and the temperature noted. From another burette 20 c.c.±0.1 of the ether-alcohol-water sample is measured into the analytical burette. This is then tightly corked and is inverted 15 times, with vigorous shaking, after which it is allowed to stand upright for at least 10 minutes, with occasional twirling between the hands, the temperature being finally reduced to the initial value. The volume of the upper layer is now read and the increase noted. A difference in temperature of 1° C. between the readings affects the result by 0.3%. The increase in volume is about 90% of the true volume of ether. The experimental data, from which the conversion curve is to be drawn, are given in Table II.

(b) *Complete analysis.*—This entails a determination of ether content as described, together with a measurement of sp. gr. at 20° C. The densities of

forty synthetic mixtures were determined, and are given for reference in Table III; from these the figures of Table IV were obtained by graphic methods. The curves of the diagram corresponding with Table IV (which does not lend itself to accurate reproduction on a small scale) give by inspection, from the ascertained ether content and sp. gr., the required alcohol content of the sample; the percentage of water follows by difference.

TABLE II.  
*Ether extraction by petroleum ether.—Volume method.*

Ether-alcohol-water, 20 c.c.; p.e., 40 c.c.; 2%  $H_2SO_4$ , 40 c.c.)

Mixtures with 94% spirit.			Mixtures with 76% spirit.		
Ether, c.c.	Gain.	Ratio.	Ether, c.c.	Gain.	Ratio.
0	1.10	—	0.38	1.02	1.04
0.98	1.04	1.09	1.86	1.76	0.95
1.96	1.86	0.95	10.00	9.16	0.92
3.90	2.76	0.92	16.20	14.72	0.90
4.62	3.68	0.91	16.40	14.70	0.90
4.16	3.78	0.91			
5.60	4.52	0.90			
6.00	5.52	0.92			
7.00	6.40	0.91			
7.94	7.25	0.91			
8.96	8.18	0.91			
9.98	9.12	0.91			
12.00	11.04	0.92			
13.98	12.88	0.90			
15.98	14.64	0.92			
16.40	14.88	0.93			
17.94	16.58	0.93			
17.96	16.68	0.93			
20.00	18.78	0.94			
26.00	18.82	0.94			

The accuracy of the method is within 1% for ether and alcohol.

*B. Vapour mixtures.*—The vapours can be completely absorbed in strong sulphuric acid, and nearly all the ether can be extracted with paraffin if the acid be then suitably diluted. If alcohol and water are to be jointly determined, the gain in weight of the acid is measured before extracting the ether; the results give the alcohol+water by difference. Where, however, it is desired to estimate each constituent, the air-stream bearing the vapours is drawn first through ignited alumina, which extracts the moisture and leaves only the alcohol and ether to be absorbed by the acid. Thus two pairs of weighings and one paraffin-extraction, made with a single sample of the vapour-laden air, give a complete analysis.

The alumina is prepared by precipitation with ammonia from aluminum nitrate; after thorough washing it is ignited at a dull red heat. The white crumbly product is very hygroscopic, and is the only material found to absorb moisture sufficiently rapidly without retaining notable quantities of alcohol and ether.

Whatever type of acid-bubbler is used should be chosen with a view to ease in pouring out as well as to efficiency and lightness; it need not have a capacity greater than about 30 c.c. One bubbler next to the alumina tube is filled with 10 c.c. of pure sulphuric acid (sp. gr. 1.84), and a second with 5 c.c. The two are weighed (unless either alone is to be determined, when neither weighings nor alumina-tubes are required) and are connected in series with the alumina-tube on one side, and with a graduated aspirator on the other through a guard-

TABLE III.

*Sp. gr. of mixtures of ether with aqueous alcohol; 20°/4°.*

[illegible]

TABLE IV.  
*Sp. gr. at 20°/4° of ether-alcohol-water.\**

Percentage of ethyl alcohol in ternary liquid.																Ether
95	90	85	80	75	70	65	60	55	50	45	40	35	30	25	20	%
787	0.801 0.783	0.815 0.798 0.779	0.820 0.813 0.795 0.775	0.840 0.823 0.809 0.792 0.771	0.853 0.838 0.822 0.805 0.788 0.768	0.865 0.850 0.835 0.819 0.802 0.784 0.766	0.877 0.861 0.847 0.831 0.815 0.799 0.782 0.760	0.889 0.874 0.857 0.844 0.823 0.812 0.796 0.778 0.756	0.901 0.887 0.871 0.855 0.839 0.824 0.808 0.792 0.775 0.752	0.913 0.899 0.884 0.868 0.852 0.836 0.820 0.804 0.788 0.771 0.749	0.924 0.910 0.896 0.880 0.864 0.848 0.832 0.816 0.800 0.784 0.767 0.745	0.934 0.922 0.907 0.891 0.876 0.860 0.844 0.828 0.812 0.796 0.780 0.763 0.741	0.944 0.933 0.920 0.901 0.888 0.872 0.856 0.840 0.824 0.808 0.792 0.776 0.755 0.737	0.954 0.944 0.930 — — — — — — — — — — 0.788 0.772 0.755 0.733	0.962 0.952 — — — — — — — — — — — — 0.751	5 10 15 20 25 30 35 40 45 50 55 60 65 70 75

\* In drawing

\* In drawing curves of these data density should be plotted against ether-content, each of the above columns forming one of the curves of the diagram.

tube containing sulphuric acid. The air-sample should contain less than 5 g. of ether, and the rate of aspiration may be as rapid as the efficiency of the absorbers will allow. It is advisable to stand the acid-bubbler in cold water. At the end of the absorption any alcohol which is present in the alumina is removed by means of a stream of dried air; the absorption tubes are then weighed (for water and for alcohol+ether), and the ether-determination is made.

For this purpose a hurette of the Mohr type is used, having a stem of 50 c.c. graduated in tenths and terminating below in a bulb of capacity not less than 80 c.c. A thermometer with cotton wool wrapping is attached, as described above. Before the analysis, mercury is put into the hurette up to the lowest mark.

The principal bubbler is now emptied into the hurette, and is washed in with the contents of the second. Enough sulphuric acid (sp. gr. 1.84) is added to bring the volume of the acid layer to 20 c.c.  $\pm$  0.1. Mercury is then run out to make room for the addition of 40 c.c.  $\pm$  0.2 of the mobile paraffin already specified, the volume of which is exactly read *in situ* after draining. More mercury is run out, and 50 c.c.  $\pm$  0.2 of water is carefully added, the hurette being tilted; a cork is at once inserted and is held firmly while the acid and water are gently but thoroughly mixed, the hurette being cooled by means of water. The hurette is now inverted 12 to 15 times, and is read in the same manner as described above. The gain in volume of the oil is from 70 to 84% of the volume of ether actually absorbed; the conversion is given in Table V, from which a curve should be drawn. Excess of alcohol vapour does not affect the ether-determination.

using acetone, the absorbing agent used for this solvent being an aqueous solution of sodium bisulphite (the Robertson-Rintoul process, E.P. 25,994 of 1901). With the development of cordite R.D.B., there arose the need for recovering ether-alcohol, another absorbent being required; and the economic value of the recoverable ether, alcohol, benzene, etc., used in other nitrocellulose and in rubber industries is great. Several methods suggested for such purpose, which have been tried on the full scale, have failed owing to fundamental unsoundness which preliminary physico-chemical work in the laboratory would have discovered.

The present paper gives a short account of two laboratory investigations dealing with the use of cresol\* and of sulphuric acid respectively, for the recovery of ether-alcohol vapours highly diluted with air; and they include a contribution to the calculation of scrubbing-efficiencies in general. The investigations relate, first, to the efficiency with which the scrubbing liquid can absorb the vapours, and, secondly, to the conditions for separating the absorbed material from the liquid.

#### CRESOL.

##### A.—Absorption.

The efficiency of absorption of a vapour by a non-volatile liquid depends in the first instance on the vapour pressures of liquid mixtures of the two. Measurements were therefore made of the vapour pressures at various temperatures of solutions in different concentrations of ether and of alcohol in cresol, pure and crude. Two experimental methods were used: in one a jacketed nitrometer (with mercury-sealed tap) and its reservoir served as a barometer into which the liquid mixture could be

TABLE V.

*Ether extraction by paraffin of b.p. 120°–140° C., from diluted sulphuric acid. Volume method.*  
*Sulphuric acid containing vapour, 20 c.c.; paraffin, 40 c.c.; water, 50 c.c.*

c.c. Ether present	..	0	0.49*	1.00	1.55	1.72†	2.00	2.46	2.98	3.74	4.96	7.07
c.c. gain of oil	..	0.00	0.33	0.76	1.10	1.31	1.50	1.93	2.30	2.92	3.99	5.85
Ratio	..	—	0.87	0.76	0.71	0.76	0.75	0.78	0.77	0.78	0.80	0.83

\*Alcohol also present 6.5 c.c.

†Alcohol also present 8.5 c.c.

A trial run may be quoted in which weighed quantities of ether, alcohol, and water were allowed to evaporate into three currents of dry air, which were united and passed through the absorbing system. The analysis gave:—

	g. per cb. m. of air.		
	Ether.	Alcohol.	Water.
Found by analysis	133	27	3.4
Actually present	134	23	3.6

Our thanks are due to the Director of Artillery for permission to publish this work, which was carried out in 1917 at the Research Department, Woolwich; and to the Superintendent of Research and Sir R. Robertson.

University College, London.  
Research Department, Woolwich.

#### THE RECOVERY OF SOLVENT-VAPOURS FROM AIR. THE USE OF CRESOL AND OF SULPHURIC ACID FOR ETHER AND ALCOHOL.

BY IRVINE MASSON AND T. LAWSON McEWAN.

The recovery of solvent-vapour from air into which it has been evaporated has for many years been in operation in the case of cordite factories

introduced; in the other, known volumes of air were drawn through the liquid in appropriate saturators, the quantity and composition of the vapour so evaporated being determined. In both methods there are inherent possibilities of error, but these can be avoided; and the results obtained were in sufficiently close agreement with each other and with the standard data by which they were tested, for all practical purposes. The effects of moisture (present in the air to be scrubbed) were also tested; the ranges of temperatures and of vapour-concentrations were restricted to those to be expected in practice.

The latent heats of solution of liquid ether and of alcohol in cresol at ordinary temperature were also measured, in order to calculate thermal effects in the scrubbers, and were found to be respectively 60 Cals. and <2 Cals. per kg. of liquid dissolving in 4 kg. of cresol.

Some of the vapour pressure data are given in Table I, corrected for the slight partial pressures of cresol, and expressed in grams of vapour per cubic metre. It will be found that, for the concentrations concerned, the curves are practically rectilinear and Henry's law holds. At higher concentrations (not recorded here) the curves first steepen and finally flatten again, as is usual with such mixtures.

It was proved that when alcohol and ether are present together in cresol, each has little or no

\* The use of cresol was the subject of a patent by Brigadier E.P. 128,640.



significant effect on the partial pressures of the other; nor, except at the higher temperatures, does the presence of dissolved water materially affect the partial pressure of ether in cresol.

TABLE I.  
Grams of vapour per cubic metre of air.

Partial press. of	g. per kg. cresol.	20°	30°	40°	50°
Pure m-cresol ..	1000	—	—	3	5
Ether + commercial m-cresol	10	7	8	9	11
	25	17	20	32	47
	50	35	44	65	93
	75	53	68	94	133
	100	71	92	123	172
Alcohol + commercial m-cresol	25	3.5	5	8	17
	50	9	11	19	29
	100	14	22	42	65

#### B.—Regeneration.

The recovery of the absorbed vapours was obviously to be accomplished by distillation; hence measurements were made of the yields got at different temperatures by distilling 1-kg. samples of 20% solutions of solvent in cresol—a strength which the vapour pressure measurements had indicated would be the maximum to be dealt with in practice. A method for analysing the distillates (described in another paper) was worked out and used.

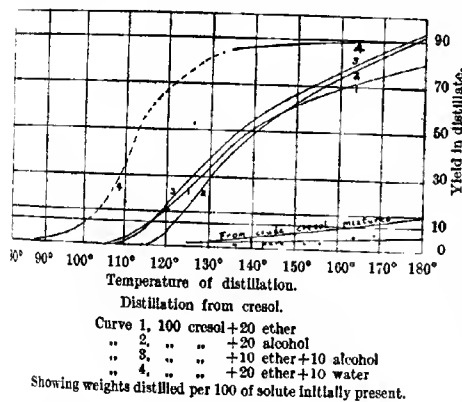


FIG. 1.

The tenacity with which cresol retains ether and alcohol, even at high temperatures, as shown by curves 1–3 of Fig. 1, pointed to the need for steam-distillation; experiments with this, recorded in curve 4, confirm the necessity and define the temperatures to be used. Finally, since cresol is itself slightly volatile, the distillates were analysed for this component; the results are shown in the lowest curves of Fig. 1.

#### C.—Application of data.

The plant initially available on the full scale consisted essentially of scrubbing towers built up of tray-segments, the vapour-laden air bubbling up through the trays in series in counter-current to the liquor, which thence passed to stills; the absorbent, now freed from solvent, was cooled and returned to the beginning of the cycle.

(a) Absorption.—The efficiency of absorption is throughout expressed as the percentage of the entering weight of vapour which is removed from the air by the scrubbing-liquid. Provided that equilibrium is established at each contact of vapour-

laden air with absorbent, the efficiency would be affected only by (1) the temperature, (2) the flow ratio, i.e., weight of absorbent used per cb. m. of air, (3) the concentration of solvent which may be already present in the absorbent, and (4) the number of "effects." Under equilibrium conditions, in a case like the present one, to which Henry's law applies, the concentration of vapour in the entering air has in itself no influence on the efficiency as defined, except through the thermal effects referred to below.

In practice, true equilibrium is not achieved in each "effect"; but no data as yet available for a tower-design suffice for the quantitative application of a theory of counter-current scrubbing (cf. Donnan and Masson, J., 1920, 236–240 r). If, however, we calculate the relationships between flow-ratio, number of effects, and efficiency, on the assumption that equilibrium is attained at each effect, we can at least establish the minimum requirements which must be met to achieve specified efficiencies; this will now be exemplified.

**Thermal effects.**—The temperature of the air-stream varies with different local conditions; that of the towers varies from top to bottom owing to the heats of absorption. The latent heat of condensation of alcohol is 250 Cals. per kg.; that of ether is 90 Cals. per kg., to which must be added about 60 Cals. for the heat of solution in cresol; and the specific heat of cresol is about 0.55. Thus if the air reaching the scrubbers contain, for example, 40 g. of alcohol and 20 g. of ether per cb. m., the heat liberated by the absorption of this could heat 1 kg. of cresol through about 23° C., or 1 kg. of cresol+1 cb. m. of air through about 15°. This figure would be increased, and the efficiency in consequence lowered, if the incoming air were richer in vapour or if the flow-rate of cresol were to be reduced. From the data about to be discussed, it appears that a rise of mean temperature of 10° would reduce the efficiency by roughly 5–10%; but the question is one of temperature-gradient and heat interchange, and is not at present amenable to more exact calculation. We may nevertheless reckon isothermally with a mean effective temperature, bearing in mind the fact that the efficiencies so deduced will approximate most nearly to the truth when the vapours are dilute and the liquor-feed is high; and by adopting here an effective temperature of 30°, we provide a margin and in a simple way obtain (as will be seen) results well borne out in practice and therefore useful as a forecast of the working conditions to be adopted.

**Efficiency of absorption.**—We may define Henry's coefficient ( $k$ ) as the equilibrium-ratio between the weight of substance dissolved per kg. of cresol and the weight remaining as vapour per cb. m. of air. For ether at 30°,  $k = 1.06$ ; hence if 1 cb. m. of ether-laden air be brought to equilibrium with 1 kg. of fresh cresol,  $1.06 + 2.06 = 51.5\%$  is the efficiency of absorption. For alcohol at 30°,  $k$  is 4.32, and the efficiency is  $4.32 + 5.32 = 81\%$ . The absorption of ether is thus the dominant problem.

In general, if  $q$  kg. of absorbent be brought into equilibrium with 1 cb. m. of air, then whether the absorbable constituent were initially all in the air, all in the absorbent, or divided between them, the final result would be that, of the total weight of it present, a fraction  $qk/(qk+1)$  must remain in the liquid. (Where Henry's law is not applicable and  $k$  is not a constant, the values of this fraction for the concentrations dealt with are found from the experimental curves.)

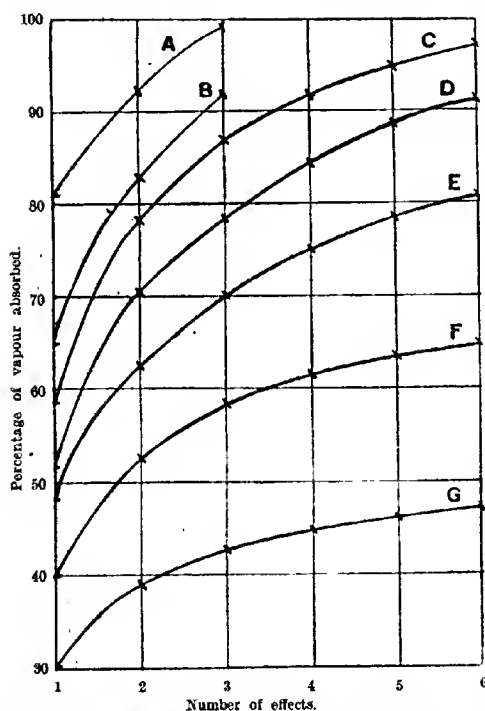
The values of  $qk/(qk+1)$  for ether at 30° and for flows of 0.4, 0.6, 0.8, 1, and 2 kg. of cresol per cb. m. of air are 30, 39, 46, 51.5, 68% respectively; with a single "effect," these numbers are the efficiencies of absorption.

Now consider a two-effect counter-current scrubber, with a specified feed of 1 kg. of cresol per

cb. m. of air, containing, say, 100 g. of ether vapour per cb. m. The first cubic metre admitted meets the layer A in the bottom effect, and 51.5% is absorbed as explained above; 48.5 g. of vapour passes on to layer B in the upper effect, which absorbs  $0.515 \times 48.5 = 25$  g.; the remaining 23.5 g. of vapour is lost. The layer A now flows out of the tower and a second cubic metre of air with 100 g. of ether meets first the layer B, now occupying the lower effect, and already charged with 25 g.; hence of the total 125 g. 51.5%, or 64.5 g., is absorbed, the remaining 60.5 g. passing up to the fresh top layer, C. Proceeding in this way for each successive admission of air and its accompanying flow of liquid, we obtain the results shown in Table II.

TABLE II.

Layer of cresol.	1st cb. m.		2nd cb. m.		3rd cb. m.		4th cb. m.		5th cb. m.	
	A.	B.	B.	C.	C.	D.	D.	E.	E.	F.
Total ether in the effect	100	48.5	125	60.5	131.5	64	133	64.5	133	64.5
Ether absorbed, g.	51.5	25	64.5	31.5	67.5	33	68.5	33	68.5	33
Vapour passing on, g.	48.5	(23.5)	60.5	(29)	64	(31)	64.5	(31.5)	64.5	(31.5)



Efficiency of absorption by cresol with multiple series effects (calculated).

A.	Alcohol, 30°	cresol feed=1 kg. per cb. m.	
B.	" 30°	" " 0.4 " "	
C.	Ether, 20°	" " 1 " "	
D.	" 30°	" " 1 " "	
E.	" "	" " 0.3 " "	
F.	" "	" " 0.6 " "	
G.	" "	" " 0.4 " "	

FIG. 2.

Thus after 4 cb. m. of air has passed, a steady state is reached, the recovery being henceforth 68.5%.

We may similarly calculate the efficiencies for other flow-rates, using the appropriate values for  $qk/(qk+1)$ , and for any specified number of effects. (General algebraic formulæ, which might replace this arithmetical method, grow too cumbersome with many effects). A separate calculation is made for

each type of system and for each different flow-rate. The results of these calculations for ether at 30° C. are shown in Fig. 2, which includes also a curve for 20° and two curves for alcohol at 30°. From this can be judged the maximum efficiency of counter-current absorption which can be reached with cresol, working either with a single effect or with multiple effects up to six, and feeds of cresol up to 1 kg. per cb. m. of air-stream.

It is clear that increasing the number of effects much beyond six is necessary only to make up for the non-attainment of equilibrium in each effect, which is in turn due to the finite velocity of dissolution of a gas, to excessive thermal effects caused by "waves" of vapour, and to imperfections in

the design of the scrubber. A feed of 1 kg. of cresol per cb. m. of air should give a recovery of over 90% with six to eight effects at an effective temperature of 30°. Experience on the large scale has justified this conclusion.

(b) *Regeneration*.—Fig. 1 is self-explanatory. It is evident that steam-distillation is called for, and that unless local rectification is provided, 3–5% of the primary distillate will be cresol, which will have to be returned to the system. One kg. of cresol containing 40 g. of alcohol and 20 g. of ether would require, for heating it from 30° to 130° with separation and distillation of the vapours, roughly 70 Cals. (specific heats of ether and of alcohol vapours = 0.44 approx.); while the remaining cresol will require to be cooled to, say, 15° before re-use, therein parting with about 65 Cals.

It is not proposed to deal with certain other factors affecting the use of cresol, such as the presence of naphthalene and of pyridine homologues in the crude material; the slight loss of cresol in the air issuing from the scrubbers, corresponding with its measurable vapour-pressure at low temperatures; its increase in viscosity after long use; and its attack on metals other than iron and steel. These were well investigated by the Research Laboratories of H.M. Factory, Gretna, where the installation of the cresol process on a large scale led to most satisfactory results.

#### SULPHURIC ACID.

The general procedure in this case was the same as with cresol, with additional work called for by two variants not paralleled in the case of cresol, namely, the strength of the sulphuric acid and the chemical action of the more concentrated acids upon alcohol.

#### A.—Absorption.

The vapour-pressure results, recorded in Table III., showed that while Henry's law is approximately valid for each strength of acid within the range of the experiments, the solubility of ether vapour in particular varies from small to great according to the concentration of the acid dealt with. The solubility-coefficients ( $k$ ) for ether range from 0.02 with 50% sulphuric acid at 30° C., to 4.2 for 82% acid at 20°.

We may thus consider the general relations between solubility-coefficients,  $k$ , as determined in the laboratory, and the efficiencies of absorption obtainable in scrubbers.

To decide the merits of an absorbent for a given vapour, we must first ascertain from the vapour pressures whether the value of  $k$  is such as will

TABLE III.  
Vapour-pressures.

% H <sub>2</sub> SO <sub>4</sub> .	Alcohol g. per kg. acid.	Pressures in grams of vapour per cubic metre.			
		20°	30°	40°	50°
81.8		Alcohol-sulphuric acid.			
	500	10	15	23.5	36.5
	250	5	8	13	17
	100	2.5	2.5	5	7
	50	0	1	2.5	3.5
20	0	0	1	2.5	
70.8	500	14	22	39	65
	250	6	7	13	17
	100	5	5	6	9
	50	2.5	3.5	3.5	5
	20	1	1	1	2.5
70.6	500	19	32	59	112
	250	10	20	31	48
	100	5	8	9.5	12.5
	50	2.5	3.5	5	6
	20	1	1	2.5	2.5
64	500	33	52	94	164
	250	9	27	53	87
	100	4	11	19	25
	50	2.5	5	9.5	11
	20	—	2.5	3.5	5
60.6	500	40	58	111	210
	250	20	30.5	59	94
	100	9	15	19	21
	50	4	8.5	7	—
	20	1	3.5	2.5	—
50.0	100	14	20	38	41
	50	6	11	22.5	36.5
	20	2.5	5	8	—
81.8		Ether-sulphuric acid.			
	Ether g. per kg. acid.				
	200	53	86	110	147
	100	18	29	46	62
	50	6	10	15	26
20	0	0	4	7	
70.7	200	138	286	485	827
	100	93	149	259	404
	50	65	78	106	154
	20	28	35	45	55
63.6*	30	53	98	167	301
	20	24	55	91	176
	10	16	22	34	70
60.0	20	117	239	—	—
	10	57	118	—	—
49.5	30	595	1180	—	—

\* This was acid which had been mixed with alcohol and subsequently distilled up to 150°—160° C.

TABLE IV.  
Values of  $qk/(qk+1)$  for different values of  $k$  and  $q$ .

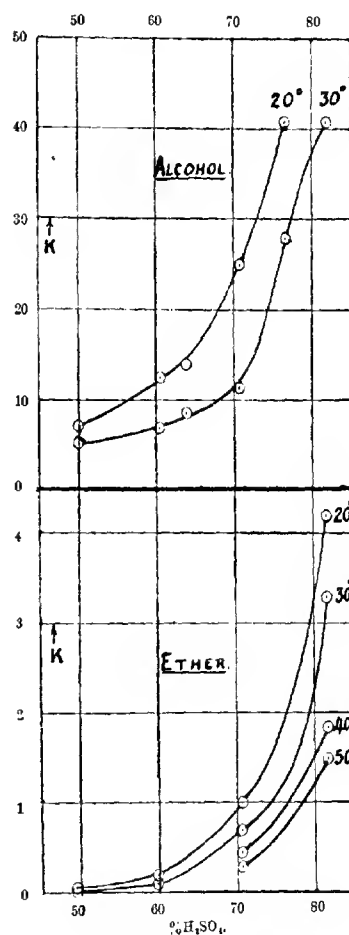
$k =$	0.1	0.3	0.7	1.0	1.5	2	3	5	10
$q = 0.1$	0.01	0.03	0.06	0.09	0.13	0.17	0.23	0.33	0.50
0.3	0.03	0.08	0.17	0.23	0.31	0.37	0.47	0.60	0.75
0.7	0.06	0.17	0.32	0.41	0.51	0.58	0.68	0.78	0.88
1.0	0.09	0.23	0.41	0.50	0.60	0.67	0.75	0.83	0.91
1.5	0.13	0.31	0.51	0.60	0.69	0.75	0.82	0.88	0.94
2.0	0.17	0.37	0.59	0.67	0.75	0.80	0.86	0.91	0.95
5.0	0.33	0.60	0.78	0.83	0.88	0.91	0.94	0.96	0.98
10.0	0.50	0.75	0.88	0.91	0.94	0.95	0.97	0.98	0.99

bring  $qk/(qk+1)$  above the critical value when the flow-rate ( $q$ ) is kept within reasonable limits. This can conveniently be ascertained from Table IV., which applies to any vapour and absorbent of the general type dealt with.

Thus, once the solubility-coefficient of the vapour in the proposed liquid has been determined, refer-

TABLE V.  
Absorption-efficiencies for specified values of  $qk/(qk+1)$  and multiple effects.

$\frac{qk}{qk+1} =$	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
No. of effects.	Efficiency of absorption.								
1	0.10	0.20	0.30	0.40	0.50	0.60	0.70	0.80	0.90
2	0.11	0.24	0.38	0.53	0.67	0.79	0.89	0.95	0.99
3	0.11	0.25	0.41	0.58	0.75	0.88	0.95	0.99	0.99
4	0.11	0.25	0.42	0.62	0.80	0.93	0.98	0.99	0.99
5	0.11	0.25	0.42	0.64	0.84	0.95	0.99	1.00	1.00
6	0.11	0.25	0.43	0.65	0.86	0.98	0.99	1.00	1.00



Solubility of vapours in sulphuric acid.

FIG. 3.

ence to Table IV., enables us to see what flow of the liquid is needed to give values of  $qk/(qk+1)$  sufficient to correspond with high efficiencies, as set forth in Table V., where the number of effects which will be required is stated. Each figure in this table is the result of a separate calculation such as that exemplified on p. 34 T.

It is seen that no really useful absorption can be expected when  $qk/(qk+1)$  is less than about 0.5, as shown to the left of the zigzag line in Table IV.

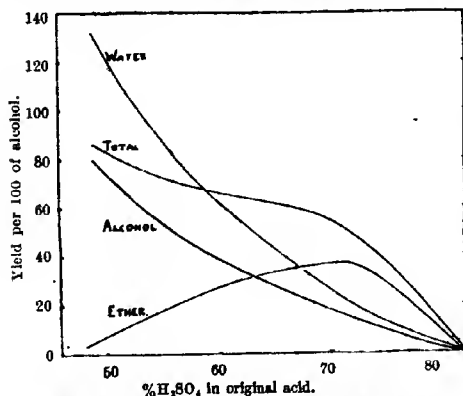
Turning now to the particular case before us, the values of  $k$ , the solubility-coefficients, are given for ether and for alcohol in Fig. 3. Reference from this to Tables IV. and V. at once shows that alcohol is very easily absorbed even in acid as weak as 50%  $H_2SO_4$ ; the coefficients are so high that difficulty would be anticipated—and is in fact met with, as will be seen—in expelling the dissolved alcohol from acid by distillation. The trend of the curves indicates that a sufficiently high value for  $k$  (alcohol) would be given by 0%  $H_2SO_4$ ; direct determinations of the partial pressure of alcohol in pure water support this, and the use of pure water-scrubbing for recovery of alcohol alone is a proved success on the large scale, being advantageous alike for efficiency and for freedom from plant troubles due to acid.

For ether, on the other hand, it is clear that without excessive feeds of liquor, no acid weaker than 70–75%  $H_2SO_4$  will result in satisfactory absorption. At an effective temperature of 30° C., the curve and tables show that a flow of 74%  $H_2SO_4$  of 1 kg. per ch. m. of air treated would give 86% efficiency of absorption, six effects being required; 1.5 kg. would give nearly complete absorption. For 66%  $H_2SO_4$ , no flow less than 5 kg. per ch. m. would give good absorption even with a large number of effects.

The remarks made in the first part of this paper concerning thermal effects are applicable here, especially in connexion with the use of the highest strengths of acid; but no measurements were made of heats of solution when the present work was done.

#### B.—Regeneration.

In absorption, ether presents the greater difficulty; in distillation, whereas ether is easily separated, alcohol remains, and if the temperature and concentration of the acid be too high, ethyl-sulphuric acid and ether can be formed, of which only the latter distils over. Experiments were therefore first made in which alcohol and sulphuric acid of various strengths were mixed in the ratio of 1:5 and were distilled evenly up to a temperature of 150° C., the distillates being measured and



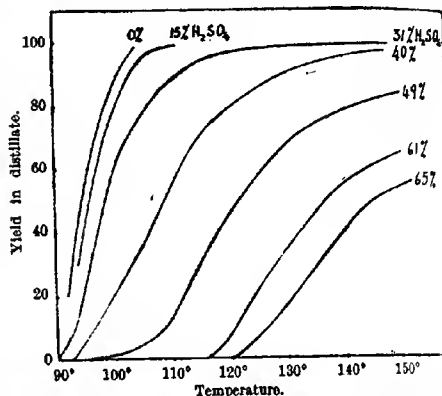
Yields on distillation to 150° C. of 100 grams alcohol + 500 grams sulphuric acid.  
(Ether is expressed in terms of alcohol).

FIG. 4.

analysed by the method described on p. 29 r. Fig. 4 shows the results obtained with acids which contained over 50%  $H_2SO_4$  before being mixed with alcohol. The various curves show the total recovery (up to 150°) per 100 g. of alcohol originally present, the separate amounts recovered as alcohol and as ether respectively, and the associated water which distils over. The ether-yield is expressed in terms

of the weight of alcohol from which it was formed during the distillation.

Considering the total recovery, it is seen that with 82%  $H_2SO_4$  practically no liquid distils over up to 150° ( $SO_2$  being evolved in considerable quantity); with 70% acid a 55% recovery is reached, of which about three-fifths appears as ether and two-fifths as alcohol; 60% acid gives 66% recovery, the proportions of alcohol and ether recovered being reversed; 50% acid gives 83% recovery, only one-twelfth now being ether. It was found that increasing the rate of heating made very little difference in the yield or composition of the distillates, nor did the presence of added ether materially aid the recovery. It is evident that to achieve proper recovery in one distillation the acid must be diluted to be at most 50%  $H_2SO_4$  (not counting the alcohol present).



Yields on distillation of 100 grams alcohol + 500 grams sulphuric acid of specific strengths (any ether formed is included in terms of alcohol).

FIG. 5.

A second series of distillations was therefore made with acids weaker than 50%  $H_2SO_4$ , the results of which, shown in Fig. 5, enable the yield on distillation to any desired temperature with specified strengths of acid, to be gauged.

The distillates obtained naturally become more aqueous the higher the yield extracted. At yields of 80%, the spirit obtained from 51%  $H_2SO_4$  is 28% alcohol; from 49%  $H_2SO_4$  it is 44%; and from 31%  $H_2SO_4$ , 55%.

Finally, there is the question of the strength of the acid after it has been subjected to distillation. With acids stronger than approximately 65%  $H_2SO_4$ , heated to 150° with alcohol, the chemical formation of ether sets free more water than is removed by distillation, so that the final acid is of about 65% strength. With weaker acids, ether-formation takes place either to a less extent or not at all, and distillation results in concentration. The net result is to give acid of 60–65%  $H_2SO_4$  at temperatures of 140°–150°, and of 50–55% at 130°.

#### C.—Conclusions as to the use of sulphuric acid.

(1) On account of the difficulty of absorbing ether, 74%  $H_2SO_4$  will require to be supplied to the scrubbers, with a flow-rate not less than 1½ kg. per ch. m. of air treated, and with at least six effects in the scrubber. Alcohol will be very thoroughly absorbed. With more concentrated acid the feed can be less, according to the tables; but it is probable that thermal effects would then outweigh the advantage gained.

(2) In the absence of alcohol, distillation of the effluent liquor at low-pressure steam temperatures will readily yield up practically all the ether; but

when alcohol is present, it is essential that the effluent be diluted to be below 50%  $H_2SO_4$ , and preferably to 40%  $H_2SO_4$  (not counting the absorbed solvent present) before it is distilled. The distillation must then be carried up to 150° for acid diluted to 60%, or to 130° with 40%, in order to regain nine-tenths of the absorbed alcohol.

(3) The acid resulting from the distillation at 150° will be about 60%  $H_2SO_4$ , or 50%  $H_2SO_4$  from that at 130°, besides containing some remaining unexpelled alcohol in the form of ethylsulphuric acid. In a cyclic recovery process, this would require to be separately concentrated up to 74% and cooled, before being returned to the top of the scrubbers.

(4) Thus it will be seen that, while sulphuric acid of about 75% strength would be of the same degree of utility as cresol if ether were the only substance to be recovered, where both ether and alcohol together are concerned, it cannot compete with cresol on account of the heavy costs attaching to the re-concentration which is required. Where alcohol alone is in question, the simplest efficient absorbent is water.

(5) In the course of the paper two tables, based on vapour pressures, are given which specify, for any simple scrubbing liquid and any gas, the minimum rates of flow and numbers of "effects" required to give stated efficiencies of absorption.

The authors' acknowledgments are due to the Director of Artillery for permitting publication; to Sir R. Robertson and Mr. A. E. Leighton for their interest in the work; and to Mr. H. Swann and Mr. H. Whittaker for their assistance in the experiments.

University College, London.  
Research Department, Woolwich.

#### DISCUSSION.

Sir FREDERIO NATHAN said that the papers gave the genesis of the work which had led to a very important step in connexion with the production of propulsive explosives. The recovery of acetone from cordite was well known, but the introduction of an explosive with ether and alcohol as the solvent involved another recovery problem. For the reasons given by the authors, and also more especially owing to the fact that it was impossible to use sulphuric acid in existing recovery plants, it had never become practicable to employ it. Fortunately the value of cresol as an absorbent of ether and alcohol had been realised, and as the result of the experiments described the recovery of very large quantities of alcohol and ether had been made possible, and in that way the cost of our explosives had been reduced.

Sir ROBERT ROBERTSON said that the earlier experiments on the absorption of ether and alcohol by sulphuric acid had shown clearly that this process was inadvisable, at least under the conditions which existed in this country. The necessary dilution of the sulphuric acid used for absorption, before it would part with its ether and alcohol, had constituted an objection, as it involved the reconcentration by heating of large quantities of sulphuric acid. This was to be avoided in the neighbourhood of propellant manufacture, on account of the possibility of fumes from the condensation apparatus getting into the danger area, which in this respect should be kept scrupulously clean. That danger, however, did not arise with the use of cresol. It was from the results of the authors' investigations that they had deduced the conditions and expressions which defined the maximum efficiency of a solvent, not only for the solvents mentioned, but also generally. For cresol the results actually obtained in practice agreed well with those predicted by this work, and in another case known to the speaker an application of the same principles to the absorption of alcohol

by water gave a result which was in good agreement as regards percentage of possible recovery with actual results obtained in both experimental and large-scale practice. The principles worked out by the authors should therefore be found useful in connexion with the absorption of vapour by solvents, the main assumption being that equilibrium between absorbent and vapour is achieved. He understood that Dr. Masson was pursuing his investigations on this subject; the absence of the data in this connexion had in the past resulted in many plants being built much larger than was necessary.

Mr. F. SPANXON said the authors had referred to the difficulties which occurred when applying his results to large-scale plant, but he had not referred to some almost equally great difficulties which occurred, particularly in the celluloid industry, before this stage was reached, namely, in obtaining a constant concentration of the solvent vapour in the air which had to be dealt with. He thought this was a more difficult problem in the case of celluloid than with cordite, for the reason that there was a larger range of thicknesses in celluloid manufacture. The thinnest material was 0.005 in., and the thickest standard material about  $\frac{1}{4}$  in. thick. With the thinnest sheets all the solvent which might be got out had gone in a few hours, and so far it had been almost impossible to find a practicable way of recovering any of this solvent. After the material was hooked and weighed there was very little left. He did not think the difficulties were so great in the case of the dope industries, because there they were dealing with large quantities of solvent in comparison with the solid residue. In the case of celluloid there was only about 10% of the raw weight to recover. Although in the celluloid industry there might be a very large number of stoves it was not possible to have one stove for every thickness, and consequently there would be a stove with half the material ready to come out and the other half still giving off vapour. Many of the patentees of methods of solvent recovery had not realised the difficulties antecedent to the actual recovery process.

Dr. W. R. ORMANDY said the authors had given in a very lucid manner the theory underlying the important practical work which M. Brégeat had done since 1916. Since then a large number of plants had been put up to work with cresol, and if the facts and figures could be obtained from M. Brégeat they would add to the value of the present paper. The most remarkable thing to him in connexion with the work was the apparent capability of cresol to absorb most of the vapours which were used industrially. Was there any connexion between the latent heat of solution and this property of the cresols to absorb a large proportion of the vapours which were present in the air? Obviously, the best liquid for the purpose was the one which absorbed the largest proportion in the liquid and left the smallest proportion in the air. It was a very interesting fact that many of the hydroxybenzene compounds, such as the thymols, which were very insoluble in water, were remarkably soluble in water which contained phenols and cresols, and this rather bore on the curious property of the cresols to dissolve so many of the vapours used industrially.

Mr. WILLIAM MACNAS said he believed that the greatest dilution of air with ether and alcohol that could be successfully treated was 5 grms. per cubic metre. In France M. Brégeat's method had been used on a much larger scale than at Gretna, but in both cases it had resulted in a great increase in the recovery. At Gretna it had been a very great success, and had saved a large amount of alcohol. He hoped before long some information would be published on the actual working of the plant in regard to which much of Dr. Masson's work referred.

Dr. Masson, in reply, said that at Woolwich they had measured the vapour pressures of a large number of volatile solvents dissolved in cresol, and in every case the results were such as led him to suppose that even volatile oils such as some of the hydrocarbons could fairly well be dealt with by cresol. That, he believed, was of importance in the rubber industry. He did not suggest that hydrocarbon oils, such as petroleum ether, could be recovered with anything like such a high efficiency as ether and alcohol, except possibly with a rather extravagant use of cresol. Nevertheless, cresol transcended all other liquids which he had tried except melted phenol, which was interesting in connexion with Dr. Ormandy's remarks. Phenol had a greater heat of reaction than cresol with ether. Mixtures of phenol and cresol, liquid at ordinary temperatures, had been considered, but for winter temperatures so much cresol was needed that no useful improvement would be gained. There were other matters, such as the slight volatility of cresol itself, which had to be taken into account, as had been found to be the case at Gretna. Other points which had been studied very thoroughly at Gretna included the increase in the viscosity of cresol when used in metal vessels. He understood that steel was almost the only metal which could be used with cresol; for instance, in one plant, in which lead-lined scrubbers had been used they were corroded through in a week, but these and various other questions had not come within his province. Nevertheless, they could rest assured that, of all the liquids hitherto examined, cresol appeared in general to be by far the best.

Meeting held at Burlington House on Feb. 7, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

### THE EROSION OF BRONZE PROPELLERS.

BY O. SILBERRAD, PH.D.

During the twelve years, 1908—1920, which these researches have occupied, notices of the principal conclusions arrived at in the early part of the work have from time to time appeared in print,<sup>1</sup> but although the  $\beta$  type of alloy to which they led has been universally adopted throughout the world for the manufacture of high-speed propellers, nevertheless no detailed or succinct record of the work has heretofore been made public; it is therefore with this object in view that the following account is now presented.

Prior to 1906 all observed cases of deterioration of ships' propellers appear to have been traceable to chemical corrosion and, with the possible exception of a few isolated cases, to have been confined to cast-iron or steel propellers. Soon after this date, however, numerous instances of deterioration occurred in bronze propellers of high-speed ships. In appearance this new form of deterioration bore a superficial resemblance to the corrosion above referred to, and it appears to have been generally assumed that chemical action was at the root of the trouble in both cases. As a result it was not until 1908, when these researches were commenced, that the phenomenon was submitted to systematic investigation.

In the meantime, as progress in marine engineering rendered greater velocities in ships practicable, the seriousness of the position became more acute, until this deterioration of the propellers was becoming the limiting factor to further developments in the direction of speed; thus, in the case of the Cunard liners, *Mauretania* and *Lusitania*, the first

set of propellers was practically destroyed after three months' running (see figs. 1—3), whilst high-speed torpedo destroyers have been known to show serious erosion in a single trial run at full speed (see fig. 4). At this juncture Messrs. Charles and P. R. Parsons, of The Manganese Bronze and Brass Co., Ltd., instigated the present researches which have resulted in the discovery of the erosion-resisting alloys that are now used throughout the world for the propellers of high-speed ships<sup>2</sup> and, incidentally, in proving that the deterioration is due to mechanical erosion brought about by a combination of frictional rub with "the action of water broken by evacuated spaces in which no air is present."<sup>3</sup>

Quite recently this subject has again been brought into prominence through the investigations of a sub-committee of the Board of Inventions and Research.<sup>4</sup> The conclusions arrived at by this Committee on the whole confirm the author's earlier observations that the deterioration is primarily due to mechanical causes. As regards chemical action, the Committee observe that there is no evidence of this,<sup>5</sup> which is also in accord with the author's investigations.<sup>6</sup> On the other hand, although the conclusions of the Committee as regards the mechanical nature of the deterioration entirely confirm the researches of the author and, indeed, throw considerable additional light on the mechanism of that part of erosion due to the collapse of evacuated spaces, nevertheless their views as to the precise nature of the underlying causes are not, in the opinion of the author, quite in accord with the observed phenomena. Prior to 1912 the author's experiments led him to the conclusion, firstly, that erosion proper is a purely mechanical effect, and, secondly, that this mechanical action was brought about by water broken by intervening evacuated spaces, or vacuum bubbles,<sup>7</sup> coupled with frictional rub of said water.<sup>8</sup>

The Committee would appear rather to hold the view that the former of these two agencies, which they aptly refer to as "water-hammer action," is operative almost to the exclusion of frictional rub: they appear to have arrived at this conclusion (a) because erosion does not appear to be the invariable result of velocity, although admittedly it is more likely to occur under the conditions pertaining in high-speed ships; (b) because erosion is observed in local patches, sometimes quite near the roots of the blades. The fact that the tips of high-speed propellers are frequently not eroded at all merely indicates that the frictional rub at these points alone is not sufficient to cause erosion. The distribution and nature of the observed eroded areas renders it perfectly obvious that the frictional rub of the water is, as a rule, only active when operating on metal which has been damaged through the collapse of the vacuum bubbles referred to above, or where local pressure and velocity are excessive.

<sup>1</sup> Eng. Pat. 232 of 1910, U.S. Pat. 551,535, Austrian Pat. 53,096, Russian Pat. 45,237, Ger. Pat. P.24,704.

<sup>2</sup> *Engineering*, 1912, 34.

<sup>3</sup> *Trans. Inst. Naval Architects* 1910, 223-247, C. A. Parsons and S. Cook.

<sup>4</sup> Cf. C. A. Parsons and S. Cook, *Trans. Inst. Naval Architects* 1910, 227.

<sup>5</sup> Editorial on the author's work (*Engineering* 1912, p. 391). "An examination of the damaged propellers showed that the entire area was quite different to ordinary corrosion in still water. In that case if dissolution takes place, it is selective in character, the metal being dissolved, a spongy mass richer in copper being left. No similar phenomena were apparent in the case of the eroded blades. Analysis showed no copper enrichment: the results agreed with the intended composition with extraordinary accuracy."

<sup>6</sup> This apt expression for the evacuated spaces was first used by Mr. Stromeyer: *Trans. Naval Architects*, 1919, 242.

<sup>7</sup> *Engineering*, 1912, 34. Editorial on propeller erosion.  
<sup>8</sup> Dr. Silberrad holds that erosion proper is a purely mechanical effect. The area attacked "in the case of the *Mauretania*" is near the hub. This was of large size, and it seems probable that there was a certain centrifugal action causing a reduction of pressure, and this region of reduced pressure was marked by the erosion. Dr. Silberrad considers that cavitation might occur in consequence, water broken by intervening evacuated spaces with no air present."



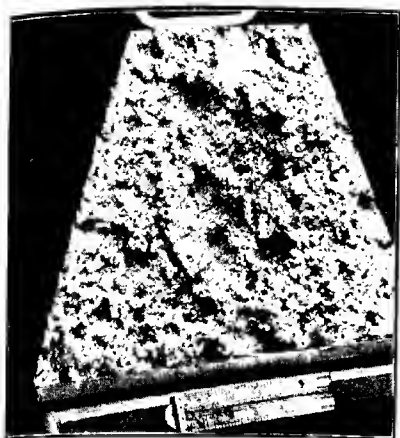


FIG. 1 ( $\frac{1}{2}$  full size).  
Part of eroded area cut from propeller of ss. *Mauretania*  
after running 3 months.

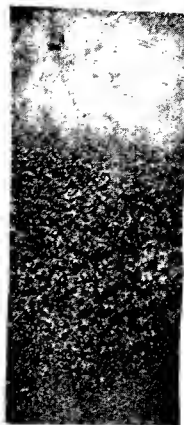


FIG. 4 ( $\frac{1}{2}$  full size).  
Part of eroded area cut from  
propeller of a destroyer after  
running 16 hours.



FIG. 6 ( $\times 2$ ).  
Bronze eroded by jet of  
high pressure water.



FIG. 2 (full size).  
on through eroded area cut from one of the first set of propellers  
of the ss. *Mauretania*.



FIG. 5 (full size).  
Valve spindle of hydraulic valve showing erosion due to frictional  
rub of water.

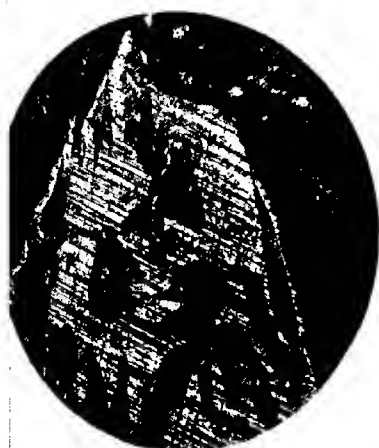


FIG. 3 ( $\times 50$ ).  
Section of eroded section showing deformed & crystals.

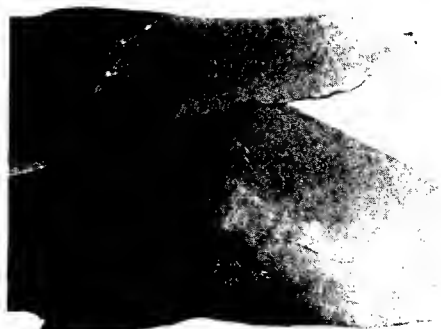


FIG. 9 ( $1/24$  full size).  
Showing 132 erosion on back.

SILBERRAD. —THE EROSION OF BRONZE PROPELLERS.



FIG. 10 ( $\times 50$ ).  
"Manganese bronze No. 1." Showing  $\alpha$  crystals embedded in  $\beta$  solid solution.



FIG. 13 ( $\times 50$ ).  
"Turbiston No. 1." Showing homogeneous  $\beta$  solid solution.



FIG. 11 ( $\times 50$ ).  
"Propeller bronze." Showing  $\alpha$  crystals embedded in a  $\beta$  solid solution.

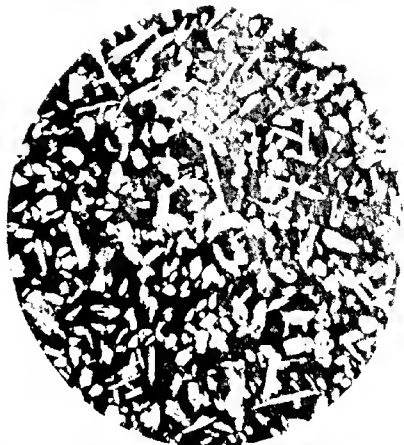


FIG. 14 ( $\times 50$ ).  
"Turbiston No. 11." Showing  $\alpha$  crystals embedded in a  $\beta$  solid solution.



FIG. 12 ( $\times 50$ ).  
"Tealodinium." Showing homogeneous  $\beta$  solid solution.

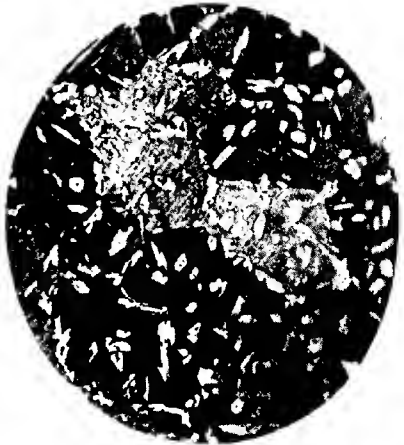


FIG. 15 ( $\times 50$ ).  
"Turbiston No. 111." Showing  $\alpha$  crystals embedded in a  $\beta$  solid solution.

That frictional rub under certain conditions can alone bring about erosion was demonstrated by the author's jet experiments carried on in 1911,<sup>1</sup> and has, in the author's opinion, been further confirmed by the experiments of the Committee themselves, which showed that not only did jets of water produce an etching effect on the surface of propeller metals,<sup>2</sup> but also that rods inserted in jets were "eroded away to a greater or less degree in parts . . . the general conclusion was that the degree of etching of these rods depended chiefly upon the duration of the experiment and the velocity of the water,"<sup>3</sup> and although the Committee's experiments were conducted with sea water, it is nevertheless fairly obvious that this action is mechanical and not chemical, for, firstly, when manganese bronze is subjected to the action of sea water it becomes tarnished all over and seldom shows any signs of etching; and, secondly, the author's original jet experiments,<sup>4</sup> which produced a similar effect, were conducted with fresh water which had been recently distilled, and which is without chemical action on manganese bronze. Hence it appears justifiable to conclude in cases where the whole of the driving face of large propellers is bright and has the appearance of being etched all over when the ship is dry-docked immediately after running at full speed, that this etching effect is in reality erosion, due very largely, if not entirely, to frictional rub. Indeed, referring to this subject in his report to the Committee, Prof. Carpenter says it "may be due to the mechanical wearing away of the metal caused by the rapid flow of water over the surface of the blade."<sup>5</sup> That it has not in general an appearance identical with the more serious patches of erosion is doubtless due to the fact that the latter is brought about by this frictional rub in combination with the passage of water broken by intervening evacuated spaces or vacuum bubbles. The difference in appearance, however, seems to be rather a question of degree: thus, where the rush of water has been extremely violent—as, for example, in the case of the spindle of high-pressure hydraulic valves, the seating of which has become so worn as to permit of the continuous passage of water (see fig. 5)—an effect is produced almost identical with that observed in the case of propellers (figs. 1–3).

As regards the influence of structure, the Committee appear to be of the opinion that there is no evidence showing "any connexion between the structure of a metal and the distribution of erosion."<sup>6</sup> It is true that the precise area attacked is determined by outside influences; nevertheless, the portion of the alloy destroyed is determined largely by the structure of the metal; indeed, frequently the substitution of an alloy of the right structure renders the propeller capable of withstanding these outside eroding influences altogether. It should, however, be observed that the Committee's investigations on this subject appear to have been confined to the examination of a propeller constructed of one of the erosion-resisting alloys which were the outcome of the earlier part of these researches, namely, an alloy of essentially  $\beta$  structure. Further, it should be observed that Professor Carpenter's work on this point does not in fact quite bear out the Committee's conclusion. Thus, he reports "that of these two constituents" (viz.,  $\alpha$  and  $\beta$  crystals) "it may be predicted that  $\alpha$  will be the more easily deformed of the two," and he actually finds two instances of such deformed  $\alpha$  crystals. He also shows that a direct application of mechanical force establishes that the  $\beta$  crystals

are not subject to distortion and that "they show that any considerable accumulation of deformed  $\alpha$  at the surface is not to be expected on account of the small amount present" (in the alloy of the new erosion-resisting type examined by him), "and, further, that such deformation must be confined to  $\alpha$  which is not entirely surrounded by  $\beta$ ."<sup>7</sup> It was precisely similar observations<sup>8</sup> which led the author in 1908 to recommend the use of an alloy of primarily  $\beta$  structure in the case of the "Mauretania" and subsequently for the manufacture of propellers liable to be subjected to erosive influences in general;<sup>9</sup> and, as Sir William Ramsay observed,<sup>10</sup> "since this alloy has been universally adopted for such purposes it must be admitted that these observations were fairly in accord with facts."

In reviewing the work of the Committee in general as set forth in Sir Charles Parsons and Mr. S. Cook's paper, though it must be admitted that much of the work merely confirms facts which have been generally accepted and utilised in the trade, the Committee have nevertheless rendered a signal service in throwing so much light on the enormous forces operative when evacuated spaces collapse under water, thus very conclusively confirming the phenomena previously observed by the author. It is, however, impossible to agree with their observation that the type of alloy used is unimportant, or that all tendency for high-speed propellers to become eroded can be eliminated through amending the design.<sup>11</sup> In the first place, the Committee only appear to have examined propellers cast of the present erosion-resisting alloys which are the outcome of the author's researches; and, further, erosion is frequently traceable to damage done to the edges of the propellers through contact with steel hawsers, wreckage, etc., namely, causes in no way bound up with the question of design. But even apart from such considerations, the difficulty in arriving at "the right design and position of propeller" for high-speed ships is such that it is obviously desirable to adopt an alloy possessed of as high a specific resistance to erosion as possible; indeed, without some such alloy it seems improbable that anything approaching the present speeds could ever have been reached.

#### 1. Investigation into the causes of deterioration.

*Chemical examination.*—At the commencement of this investigation the idea that the deterioration in question was due to chemical action was so generally prevalent that it was difficult to approach the subject with a truly open mind; a careful chemical examination of the damaged surfaces in a large number of cases, however, entirely failed to supply any evidence of chemical action; there was no sign of any concentration of copper or deficiency of zinc, as would be expected in the event of such severe deterioration being the result of chemical action. As regards composition the alloy proved to be the ordinary high-tension manganese bronze in all the early cases examined; the only possible indication of chemical action at all lay in the observation that the crystalline structure of pro-

<sup>1</sup> *Trans. Inst. Naval Architects*, 1910, 233.

<sup>2</sup> *Engineering*, 1912, 33. Editorial on propeller erosion.

<sup>3</sup> *Parsons & Silberrad*, Eng. Pat. 232 of 1910. In this connexion it is interesting to note that two years later, viz., in 1912, the *Gewerkschaft Rubelbrönze* of Berlin recommended the use of an alloy of  $\beta$  structure as being suitable for "propellers of battleships and other high class steamships," alleging that this bronze was identical with the "bronze C" manufactured by them in 1908. It should, however, be observed that the physical properties of the alloy recommended by them in 1912 do not coincide with those published in respect of their alloy of 1908, with which the alloy they subsequently recommended was supposed to be identical; and, further, that on the strength of this the *Gewerkschaft Rubelbrönze* of Berlin attacked the Austrian equivalent of this patent (Austrian Pat. 53094 of 1910 by Parsons & Silberrad), but were unsuccessful in their suit in spite of the fact that this country was at war with Austria at the time.

<sup>4</sup> *Sir William Ramsay*, *Engineering*, Nov. 21, 1913.

<sup>5</sup> *Trans. Inst. Naval Architects*, 1910, 247.

<sup>6</sup> *Engineering*, 1912, 33.

<sup>7</sup> *Trans. Inst. Naval Architects*, 1910, 235.

<sup>8</sup> *Trans. Inst. Naval Architects*, 1910, 226.

<sup>9</sup> *Engineering*, 1912, 33. Editorial on propeller erosion.

<sup>10</sup> *Trans. Inst. Naval Architects*, 1910, 232.

<sup>11</sup> *Trans. Inst. Naval Architects*, 1910, 232.

propellers was frequently clearly observable over considerable areas, the surface being bright and having the appearance of recent etching when the propellers first come out of the water. It seems probable, however, that this etching also is due to mechanical erosion, for the above phenomenon was only noticeable providing the ship had been running at full speed immediately before dry-docking; the propellers were always observed to be tarnished all over if she had been at rest two or three days previously. Experiments with a view to reproducing this condition were therefore undertaken, and it was found that a precisely similar appearance can be produced by subjecting the alloy to the action of a jet of high-pressure water, whereas on immersing a piece of manganese bronze in sea water it became tarnished rather than etched. Fig. 6 represents a specimen which has been subjected to such a high-pressure jet; the best results were obtained with small direct jets constructed of steel and supplied with water from inverted steel cylinders into which nitrogen at 2000 lb. per sq. in. was forced by means of a three-stage compressor, the water being covered with a layer of B.P. petroleum to prevent solution of the nitrogen.

any electric leak detectable throughout any of the ships examined.

The suggestion made by Mr. Ramsay<sup>1</sup> that local electric disturbances set up through mechanical stresses might be the cause of the deterioration led to the examination of the behaviour of a sample of this same alloy when submitted to a variable stress under sea water. This experiment was carried out by submerging a bar  $8 \times \frac{1}{2} \times \frac{1}{4}$  in. in sea water, one end of the bar being held rigidly and the other end attached to an eccentric making 4 strokes of 1 in. per minute. The results are incorporated in Table I., below, from which it will be observed that the alteration in weight due to corrosion in this case is only very slightly greater than that suffered by a similar bar of this alloy not under stress; and that in both cases the loss is entirely negligible as a factor in dealing with the severe type of deterioration under consideration.

*Physical examination.*—Although the above observations were sufficient to constitute fairly conclusive evidence that the deterioration was not due to chemical or galvanic action, they are nevertheless all of a negative rather than a positive nature; it was therefore decided to make full examination

TABLE I.  
Action of sea water on propeller alloys.

Duration of experiment in hours.	Loss of weight in g. per sq. metre exposed.					
	Standard manganese bronze (cut from one of <i>Mauretania's</i> first propellers) not stressed.		Standard manganese bronze (cut from one of <i>Mauretania's</i> first propellers) stressed.		Standard turbadium, unstressed.	
	Total loss during experiment in g. per sq. m. exposed.	Mean loss per 4 hrs. in g. per sq. m. exposed.	Total loss during experiment in g. per sq. m. exposed.	Mean loss per 24 hrs. in g. per sq. m. exposed.	Total loss during experiment in g. per sq. m. exposed.	Mean loss per sq. m. in g. per sq. m. exposed per 24 hours.
24	0.349	0.349	0.362	0.362	0.16	0.160
48	0.600	0.300	0.611	0.306	0.31	0.155
72	0.848	0.283	0.860	0.287	0.46	0.153
96	1.150	0.288	1.176	0.294	0.60	0.150
120	1.402	0.280	1.424	0.285	0.76	0.152
144	1.700	0.283	1.703	0.294	0.91	0.151
168	2.036	0.291	2.034	0.291	1.07	0.153
192	2.322	0.275	2.248	0.281	1.20	0.150
216	2.621	0.280	2.560	0.284	1.36	0.151
240	2.780	0.278	2.822	0.282	1.50	0.150

In order to investigate further the extent to which chemical action plays a part, a sample of manganese bronze cut from one of the wing propellers of the s.s. "*Mauretania*" was submitted to the action of sea water, the alteration in weight per sq. m. exposed being recorded from day to day. The results are given in Table I., from which it will be observed that the average loss of weight of this particular bronze was 0.28 g. per sq. m. per day. The maximum depth to which the deterioration had reached in the particular propeller from which this sample was cut was  $2\frac{1}{4}$  in., from which it will be observed that it would take over 5000 years<sup>2</sup> to effect the observed deterioration by chemical corrosion; and since the deterioration in question was caused in 3 months it becomes self-evident that chemical corrosion is a negligible factor in the deterioration under consideration.

*Galvanic action.*—No evidence of galvanic action being a factor could be obtained. In all the most pronounced cases examined the protecting zinc plates in the neighbourhood of each of the propellers were in perfectly normal condition, nor was there

of a series of damaged propellers with a view to gaining a further insight into the nature of the operating causes.

*Micro-examination* of the eroded surfaces of a large number of propellers revealed distinct evidence of deformed  $\alpha$  crystals; this was confirmed by cutting pieces from the eroded portions, embedding them in soft solder, and examining them after sectioning and polishing.

In the early days of these researches the propellers examined possessed the normal structure of manganese bronze, namely, essentially an  $\alpha$ -structure. In general these samples were remarkably similar and showed deformed  $\alpha$  crystals wherever these predominated; cases were also observed of part of the alloy having been washed away, leaving protruding crystals, most of which showed varying degrees of deformation. In a few isolated cases where the proportion of zinc in the alloy had been allowed to get a little above normal the alloy assumed a mixed  $\alpha$  and  $\beta$  structure; in these cases it was observed that the  $\beta$  crystals stood up to the erosion remarkably well, whereas the  $\alpha$  crystals, where they were not embedded in  $\beta$ , had either been crushed, broken off, or else washed right away. A typical example of such an eroded

<sup>1</sup>  $10,000 \times 2.5 \times 2.54 \times 8.821 \div 0.28 \times 365.25 = 5100.8$  years.  
<sup>2</sup> *Engineering*, May 24, 1912.

edge is illustrated in fig. 2, the propellers in question having been scrapped after running less than three months on account of erosion. In this connexion fig. 3 is also interesting as illustrating a crystal which has been deformed and broken at the point.

propeller blades. A thorough examination of a very large number of propellers, however, rendered it possible to arrive at a series of generalisations as to the areas attacked and to classify these into three types, each type being traceable to special conditions productive of currents of water, broken

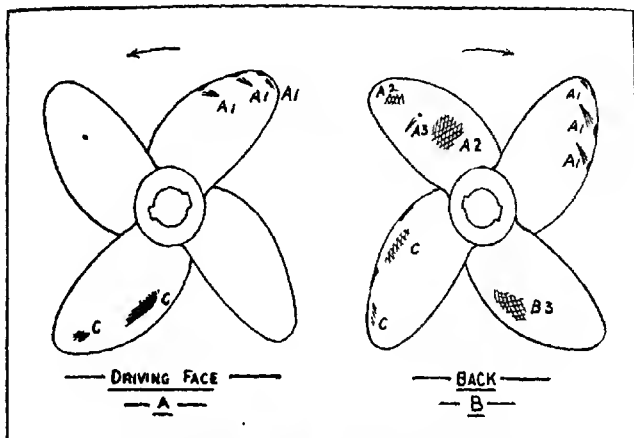


FIG. 7.—(1/100 full size.)  
Diagram of 4-bladed propeller showing various types of erosion.

It will be observed therefore that micro-examination of eroded bronze revealed distinct evidence as to the deterioration being mechanical in origin, and also as to the superiority of  $\beta$  crystals in their capacity to withstand this erosion. These conclusions were subsequently very fully confirmed by taking small samples of a very large series of propellers as they were cast, submitting these samples to micro-examination and subsequently inspecting the propellers from time to time as they developed any defect in use. In this way so close a connexion between the microstructure and the capacity to withstand erosion under any given conditions was established that it became possible by merely studying the microstructure of any given propeller to give to it a relative figure of merit which was closely indicative of its capacity to withstand erosion.

**Macro-examination.**—An exhaustive macro-examination of a large number of eroded propellers was next entered upon, which established, in the first place, that dirt in the castings played little or no part as a determining factor, either as to the position or degree of the erosion produced. In this connexion many cases of unsound castings were examined, and although instances remarkably similar to erosion were found, when propellers having such defects were put to work the cavities in question showed no signs of developing into erosion; indeed, if anything, they assumed a slightly smoother appearance.

In the second place, it was noted that eroded propellers belonged to one of two classes—they were either relatively small propellers running at a high speed (i.e., 600–800 r.p.m.), as in the case of torpedo destroyers; or else they were large propellers transmitting great power, as in the case of battleships or fast liners, as, for example, the "Mauretania," the "Lusitania," the "La France," and the "Imperator."

As regards the areas eroded in the different propellers examined, these were most varied and in the first instance most perplexing, erosion being observed on the boss, base, centre, and edges of the

by evacuated cavities, or vacuum bubbles, impinging on the areas eroded.

#### Classification of the various types of erosion.

##### Type A. Erosion due to damage done to the propeller.

Erosion of this type divides itself under three heads:—

**Type A. 1. Erosion due to damage done to the leading edge.**—Whenever the leading edge of a high-speed propeller is bent or deformed an eroded area results on that side of the blade towards which the damaged edge has been deflected. Thus the erosion starting from damage of this sort will be found on the driving face if the leading edge has been bent towards that face (see fig. 7 A), whereas if the damage done to the leading edge has caused the metal to become bent backwards, the erosion will appear on the back of the propeller (see fig. 7 B). Frequently these areas of erosion are deep and very clearly defined, often tailing off in comet-shaped grooves. It thus becomes evident that the contributing conditions which lead to erosion of this type are as follows:—

The leading edge being distorted leaves a trail of water broken with evacuated spaces starting from the distortion on the side towards which the blade has been deflected; as this current of broken water sweeps over the blade the vacuum bubbles therein collapse, and the combined frictional rub and water-hammer action so produced cause the erosion observed. As is to be expected, erosion of this type is generally situated on an arc described from the centre of the propeller and passing through the damaged edge; where exceptions have been observed these appear to be due to deflecting currents influencing the flow of the broken water caused by the damaged edge.

**Type A. 2. Erosion due to damage done to the following edge.**—Deformation of the following edge of a blade is liable to cause a stream of broken water to impinge on the back of the next blade of the same propeller (fig. 7 B). Erosion of this type occasion-

ally covers a considerable area, but is seldom deep; its origin is attributable to causes precisely analogous to those which bring about the A. 1 variety—the distorted following edge leaves a trail of water broken with evacuated spaces which impinges on the back of the next blade; the current in this case is generally more disturbed and, as a consequence, the erosion is less intense but covers a greater area.

**Type A. 3. Erosion due to holes in the propeller** (as when the plug comes out of the shackle hole). Under these circumstances a comet-shaped area of erosion results on the back of the blade (fig. 7a), the eroded area being on an arc described from the centre of the propeller and passing through the hole. A little consideration will render it obvious that erosion of this type is due to the following causes:—

The water on the driving face of the propeller being under pressure rushes through the shackle hole, thus taking up the rotary movement of the propeller; the jet of water so produced strikes the relatively stationary water behind the propeller, thus causing cavitation in the form of an evacuated space round the jet; the broken water so produced is subsequently swept over the back of the propeller on which the vacuum bubbles collapse, causing the comet-shaped eroded area invariably observed when the plug comes out of the shackle hole of a high-speed propeller.

**Type B. Erosion due to disturbances caused by the propeller itself.**

Three distinct types of erosion arising from disturbances set up by the propeller itself have been observed, namely—

**Type B. 1. Erosion due to high number of revolutions made by the propeller.**—When a propeller of any given design is running at too high a speed erosion appears at or near the root of the driving face (fig. 8). In these cases it would seem that erosion is due to the centrifugal action caused by such rapid revolution, as a result of which water,

broken by evacuated spaces, is whirled outwards into areas of greater pressure, thus causing the vacuum bubbles to collapse on the surface of the blade near the root.

**B. 2. Erosion due to stream lines caused by the following edge of the preceding propeller.**—Erosion of this type generally appears as a deep cut on the boss near the back of the blade following, and seems to be confined to propellers with very wide blades having a relatively small diameter and making a fairly high number of r.p.m. (See Fig. 9.)

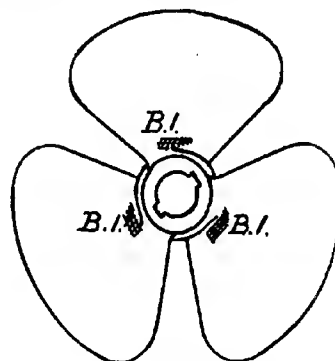


FIG. 8.—(1/25 full size.)  
Diagram of 3-bladed propeller showing B1 erosion on driving face.

**Type B. 3. Erosion due to backwash from the sides of the ship.**—This is liable to occur where the wing propellers have insufficient clearance, especially if they turn inwards; in this case the backwash causes broken water to be thrown on to the propellers, thus producing severe erosion (fig. 7a). Figure 1 illustrates an extreme case of this class, in which erosion to the depth of 2½ in. was caused

TABLE II.  
Classification of causes of erosion.

Class.	Cause.	Position and appearance.	Illustration.	Remedy.
<b>A.—Erosion due to damage done to propeller.</b>				
1	Deformation of leading edge— (a) Blade distorted towards driving face.	Comet-shaped groove on driving face starting from deformation.	A <sub>1</sub> , fig. 7A.	Avoid contact with hawsers, wreckage, etc. To repair damage trim deformed edge down to the level of surrounding metal.
	(b) Blade deformed towards back.	Comet-shaped groove on back starting from deformation.	A <sub>1</sub> , fig. 7B.	Do. do.
2	Deformation of following edge.	Area often ill-defined but generally shallow on back of the following blade.	A <sub>2</sub> , fig. 7B.	Do. do.
3	Shackle hole being unplugged.	Comet-shaped area on back of blade starting from shackle hole.	A <sub>3</sub> , fig. 7B.	Keep shackle hole plugged.
<b>B.—Erosion due to disturbances caused by the propeller itself.</b>				
1	Propeller of given design running at too high speed.	Clearly defined patch of erosion, generally deep, situated at or near root of driving face.	B <sub>1</sub> , fig. 8.	Run propeller slower, using, if need be, geared turbines and larger propellers.
2	Unsatisfactory design of following edges.	Deep cut-like erosion on boss near root of back of propeller blades.	B <sub>1</sub> , fig. 9.	Alter design of propeller, particularly following edges of the blades.
3	Backwash from side of ship due to insufficient clearance or propeller turning inwards.	Area often considerable and sometimes deep; generally situated on back of propeller about half way up and towards following edge.	B <sub>3</sub> , fig. 7B.	Give wing propellers plenty of clearance and arrange for them to turn outwards.
<b>C.—Erosion due to influences external to the propeller.</b>				
1	Stream lines of water broken by evacuated spaces produced by a propeller forward of eroded propeller.	On both sides of propeller generally about half way up and towards leading edge, erosion often being deep, especially on driving face.	Fig. 7A.	Alter relative arrangement of propellers.
2	Stream lines of water broken by evacuated spaces produced by "A brackets" forward of eroded propeller.	do. do.	Fig. 7B.	Alter design and arrangement of "A brackets" supporting propeller.
3	Stream lines of water broken by evacuated spaces produced by the ship itself.	do. do.		Alter design of ship.



in three months. As regards position, this type of erosion is generally to be found on the back of the blade, though naturally it varies with the precise position of the seat of the cause.

*Type C. Erosion due to passage of some body forward of the propeller*, which causes a stream of water broken by evacuated spaces to impinge on the propellers. Erosion of this type is generally due to one of the following causes:—

*Type C. 1. Stream lines of water broken by evacuated spaces produced by another propeller forward of the propeller in question.*

*Type C. 2. Stream lines of water broken by evacuated spaces produced by "A brackets" situated forward of the propeller eroded.*

*Type C. 3. Stream lines of water broken by evacuated spaces produced by the ship itself.*

Such erosion generally appears on both sides of the propeller and varies enormously both in shape and intensity. It occurs on the back whenever a stream of water broken by evacuated cavities impinges on the propeller and is due to the collapse of these evacuated spaces on striking the surface in question (fig. 6a). Similar erosion on the driving face is obviously due to the same cause; the streams of broken water on being cut by the propellers suddenly enter an area of high pressures with the result that the evacuated spaces collapse on the driving face at a distance from the leading edge dependent on the peripheral velocity of the propeller at the point of cutting. As a general rule erosion due to this cause will therefore be found on both sides of the propeller, but is likely to be more intense on the driving face than on the back.

For the sake of convenience in reference these various types of erosion have been classified in Table II, under the causes to which they are attributable.

#### PART II.—Production of propeller alloys possessing relatively high specific resistance to erosion.

From the investigations described above the following conclusions may be drawn respecting the nature of this deterioration:—

1. Chemical action is a negligible factor.

quently collapse on the propeller blades, both of which conditions, it will be observed, are more liable to arise in high-speed ships or propellers moving at a high velocity.

It has also been established above that where alloys containing  $\alpha$  crystals are exposed to this action, these  $\alpha$  crystals are liable to deformation unless they are wholly embedded in  $\beta$  crystals, from which it would appear that a bronze composed essentially of a  $\beta$  structure is best calculated to withstand the deterioration under consideration.

At the date when these researches were commenced, however (1908),  $\beta$  alloys consisting essentially of copper and zinc were regarded as being mechanically unsound; this research therefore next turned upon the problem of discovering suitable additions to copper-zinc alloys of the manganese bronze type which would change them from essentially  $\alpha$  alloys to those of  $\beta$  structure without impairing their physical properties in any way. In carrying out this part of the investigation use was made of Guillet's work on complex brasses,<sup>22</sup> wherein he showed that one part of zinc may be replaced by an equivalent quantity of any of a series of certain other metals without affecting the structure of the original copper-zinc alloy. These "equivalents" must not in any way be confounded with the atomic weights of the metals, to which they do not appear to bear any very simple relationship. Thus, 1 part of zinc is equivalent to aluminium 0.16 part, iron 1.1, manganese 2.0, cadmium 1.0, lead 1.0, silicon 0.1, or tin 0.5 part. Making use of these equivalents a large series of alloys possessed of  $\beta$  structure was made; in each case part of a 300-lb. charge was cast in sand and part in chill; test pieces were cut from each, and the alloy thoroughly examined both mechanically and microscopically. As this paper deals essentially with erosion and does not pretend to go into metallurgical problems it is not proposed to give these researches in detail; the following table, however, sets forth a few of the more important results which led to the production of a mechanically sound alloy of  $\beta$  structure, and gives the composition and properties of a series of alloys representative of each step in the research.

TABLE III.  
Development of bronzes.

Composition of alloy.			Composition of corresponding imaginary brass.		Mechanical properties.			
Copper. %	Zinc. %	Additional metals.	Copper. %	Zinc. %	Tensile strength, tons per sq. in.	Elastic limit, tons per sq. in.	Elongation, %	Hardness, Brinell number.
59.0	47.0	—	53.0	47.0	17.2	2.5	16	82
53.5	45.5	Sn. 1%	53.0	47.0	Unworkable.		—	200
53.0	46.0	Mn. 1%	53.2	46.8	27.5	5.1	22	98
50.5	40.5	Mn. 9%	53.4	46.6	35.0	12.7	19	90
59.0	39.0	Al. 2%	53.6	46.4	25.5	20.4	12	100
55.16	43.0	Al. 0.27, Mn. 4.123, Fe. 1.44%	53.5	46.5	30.0	16.5	25	—
Standard manganese bronze for comparison					33.0	17.0	20	120

All these alloys possessed a homogeneous  $\beta$ -structure with the exception of the standard manganese bronze, which showed strong  $\alpha$ -crystallisation. The above figures show that tin in appreciable quantity appears to act very detrimentally on  $\beta$ -alloys. The marked improvement in physical properties caused by the introduction of manganese should be noted, though the presence of as much as 9% renders the alloy troublesome in the foundry. The chief effect of aluminium appears to be to raise the elastic limit. The alloy containing Al, Mn, and Fe may be regarded as the parent of the present erosion-resisting bronzes. The first propellers made of it were cast by The Manganese Bronze & Brass Co. in 1903, for the *Maurania*. The first alloy of the type of standard manganese bronze was introduced by Mr. P. M. Parsons of The Manganese Bronze & Brass Co. in 1874.

2. Galvanic action does not appear to come into play at all.

3. The presence of dirt in the costings has only a slight detrimental effect.

4. The true cause of the deterioration is mechanical and is therefore properly termed erosion, the determining factors being:—

- (a) The frictional rub of the water.
- (b) The impinging on the propeller blades of water broken with evacuated spaces which subse-

From these results it will be observed that although a copper-zinc brass of purely  $\beta$  structure is not a satisfactory alloy, nevertheless alloys quite equal in mechanical properties to the old-established  $\alpha$  bronzes can be produced by introducing small quantities of aluminium, manganese, iron, etc. Having thus established the practicability of producing improved erosion-resisting alloys of this

<sup>22</sup> Guillet, *Rev. de M.A.*, 1906, 159.

type, their further development and application followed as a matter of course, and such alloys are now used throughout the world for the manufacture of propellers for high-speed ships. Photomicrographs of some of these alloys are given in figs. 10-15. In No. 10 (tensile strength 34 tons, elastic limit 17 tons per sq. in., elongation 25%) the  $\alpha$  crystals are sufficiently protected to render the alloy immune from any but the most severe erosive influences to which marine propellers are subject, whilst the  $\alpha$  crystals present render the alloy much more easily repaired or worked than a homogeneous  $\beta$  alloy. Similar remarks apply to a lesser extent to No. 11 (t.s. 30, el. lim. 15.5, elong., 33%), which although essentially an  $\alpha$  bronze still has sufficient  $\beta$  crystallisation for protective purposes where the eroding influences are not excessive. Nos. 12 and 13 (t.s. 40-42, el. lim. 20, elong. 17-25%) represent a homogeneous  $\beta$  solution and have the highest possible specific resistance to erosion. No. 14 (t.s. 38-39, el. lim. 18.5, elong. 30-35%), and No. 15 (t.s. 33-35, el. lim. 17.5, elong. 25-30%) have  $\alpha$  crystals embedded in  $\beta$  and are excellent all-round propeller alloys. It is noteworthy that although the former of these alloys contains a higher percentage of zinc, it is even less subject to corrosion than manganese bronze (see Table I.); it is suggested that this may be due to the extreme homogeneity of the alloy, which is a practically pure  $\beta$  solution.

The first ship equipped with propellers cast of an alloy of the new type was the "Mauretania." It will be remembered that in three months this ship wore out her first set of propellers, which were cast of the old type of bronze (see fig. 1); the propellers of the new alloy were fitted in January, 1909, and it is interesting to note that the Surveyor for the Germanische Lloyd, on inspecting these new propellers after they had run for six months—i.e., twice as long as the original ones of normal manganese bronze lasted—reported that they were in perfect condition, there was no sign of wasting or erosion, and the surface appeared to be as good in every way as when the castings were first made.

In conclusion I desire to express my thanks to Mr. P. R. Parsons for his most skilful co-operation throughout these researches. My thanks are also due to Sir Phillip Watts, K.C.B., F.R.S., for his valued advice on numerous occasions, to Dr. Deach for his assistance in connexion with the photo-micrographic records, to Mr. A. T. Quelch, O.B.E., Mr. F. G. Martin, The Manganese Bronze and Brass Co., Ltd., Messrs. J. Stone and Co., Messrs. Billington and Newton, Ltd., the Superintendents of the various Admiralty Dockyards, The Cunard Co., Ltd., Messrs. Harland and Wolff, Messrs. Cammell, Laird and Co., Ltd., and Messrs. Alfred Holt and Co., Ltd., for the facilities afforded me on all occasions during the last twelve years when inspecting eroded propellers and otherwise pursuing these investigations.

#### DISCUSSION.

Mr. A. T. QUELCH said that during the past thirteen years he had made 16,000 or 17,000 tons of bronze propellers and propeller blades; a large number of these had been made with the  $\beta$ -alloys referred to in the paper, and he could concur with the author that the erosion was purely mechanical. He had had occasion to examine very many high-speed propellers. In the case of the "Swift"—a very fast scout type of destroyer, designed for 36 knots—difficulty had been found in keeping up the speed for the six hours full-speed trials. Experiments with the design of the propellers had been started. Altogether 26 propellers had been made for that ship, and examination of these had revealed the very curious way in which, as the design was altered, the area of the eroded part moved about, a fact which proved that the erosion

was purely mechanical. In that particular case the expanded area of the three blades was actually greater than the disc area, which fact introduced that peculiar cause of erosion, the interference of one blade with another, which produced a deep cut on the back. During the war the greater number of the destroyers built had been built of the geared turbine type, giving reduced speed of the propellers, in the hope that it would put an end to erosion troubles. Soon afterwards, however, serious erosion had been reported to be taking place on the Bruce-Douglas type of destroyer leader. Examination had shown that the greater proportion of this so-called erosion was dirt washed out of the propeller, due to a great extent to the design of the propeller. Unfortunately, the engineer often did not consider whether the designs were good or bad from the founder's point of view. In this particular case the propellers were of such a large surface, coupled with a short boss, that both the leading and following edges of the blades overlapped the top and bottom of the boss. Propellers were cast with the driving face downwards, so that the following edge of the blade was considerably below the level of the bottom of the boss. The gate was put into the bottom of the boss so that the first flow of metal which entered the boss had to run down hill, and there was a considerable disturbance of this metal by eddies etc., and the production of a large amount of oxide. The section of the blade of those particular propellers, about six inches from the tip, was about 8 ft. long by about 1½ in. thick, so that the dirt had not much chance to get away. The only serious erosion in this particular case was on the back of the blade, almost on the fillet, and it was due, in his opinion, to the very badly designed A bracket. The section of this A bracket was very square, and, in addition, there was a lubricator in the angle of the A bracket, by the boss, which had the effect of throwing a jet of disturbed water on to the propeller, with the result that grooves were eroded in the blade to the extent of 1½ in. deep.

He would like to ask the author as to the size of crystals in the  $\beta$  structure which could be produced by adding various metals, e.g., Al, Mn, Fe, to the ordinary copper-zinc alloy. There was considerable variation in the size of the crystals, although their general type was the same, and this might have a very considerable influence on the mechanical properties. Whereas quite favourable results might be obtained in an ordinary tensile test piece, when the alloy was exposed to the stresses obtaining in the case of a propeller, in which there was considerable variation in stress and reversals of stress at different times, there might be failure in the material in resisting fatigue, which was most important with any running machinery. He had had very little trouble with the  $\beta$ -alloys, in which he had been particularly interested, but he believed certain troubles had occurred in propellers using alloys of somewhat similar structure where, for no apparent reason, the whole of the blade had broken off. He had recently manufactured some large propellers for Japanese battleships, of turbidinium, which weighed, in their finished state, 13½ tons each. One test-piece had been taken from each blade and one from the boss. The elastic limit of all the test pieces actually taken from the first of these propellers was 21 tons, the ultimate strength averaged 41 tons, and the average elongation 20%, which was a good result with a copper-zinc alloy. In the case of these alloys there was not the breakdown at the yield point that there was in steel, but the elastic limit was defined as such a stress as would produce a permanent set of 0.003 in. Since making these propellers he had made a smaller propeller of a  $\beta$ -type of alloy, but arrived at in a different manner from turbidinium, the object being to get a little

more stretch or ductility in the alloy. In this case the test piece taken from the propeller showed an elastic limit of 18 tons, an ultimate strength of 38 tons, and 27% elongation, with a cold bend test on a 1 in. square bar between 12 in. points of support of over 90° without fracture. With regard to the propellers of the "Manretania" and "Lusitania," he thought they were inclined to give a little too much credit to the alloy. The original propellers on both these vessels had been of the built-up type, i.e., the blades had been cast separately and bolted on to the boss, so that the external conditions would be rather worse than in the solid propeller. The design of solid propellers eventually adopted was very different, and it was a little difficult when comparing the results in resisting erosion to say how much credit was due to the alloy and how much to the altered design of the propeller. In the case of the Tribal class of destroyers built about 1910, propellers had been made of exactly the same design, some of ordinary manganese bronze and some of turbadium, and the great advantage in having the  $\beta$ -type of alloy, from the point of view of resisting erosion, had been evident. There was still some slight erosion, but it was very difficult to produce an alloy which would have the necessary mechanical properties to withstand the arduous service of a propeller in constant work, where there were very many stresses introduced, especially in the case of light-draught boats, like destroyers, and at the same time be the best structure to resist erosion. In the case of the "Hood," for instance, the finished weight of each propeller was about 19½ tons, and in the actual manufacture 30 tons of metal had to be dealt with. It would therefore be realised what a very big problem it was to produce an alloy which would meet all the mechanical tests and at the same time satisfy the very arduous conditions of propeller running and also offer a high resistance to erosion.

Mr. H. TALBOT said that the author had brought out two points which, in his (the speaker's) opinion, were almost conclusive evidence in favour of the mechanical explanation. The first was the existence of what was practically a critical velocity of erosion, above which the rate of deterioration was especially marked; the second was the fact that equally resistant alloys contained metals of the same chemical family in proportions which had no connexion with their chemical combining weights.

Dr. SILBERRAD, in replying, said that he quite agreed with the observations respecting the influence of altering the shape and design of any given propeller on the position and nature of erosion produced; indeed the observance of such cases had contributed very materially to the classification of the different types of erosion. With regard to the tendency of homogeneous  $\beta$ -alloys to solidify in large crystals, very little seemed at present to be known about the particular combinations which influenced this. Cases where alloys on solidification formed crystals as much as two inches long and several pounds weight had come before his notice, and in such cases, as a rule, a very slight alteration in composition had been found to cause the alloy entirely to change its properties in this respect. In that case the alloy was very easily broken, not with one single hit, but when subjected to continuous vibration. One of his exhibits was a sample from a propeller made of an alloy which showed this tendency; it had broken off short in use. By taking Guillet's equivalents it was possible to predict the influence of various metals on the alloy to some extent and thus obtain satisfactory mechanical results. He was never able, however, to get any idea at all as to what effect an alteration in composition would have on the tendency to form large crystals. That appeared to resolve itself into a question of trial and error, and the experiments ran into hundreds before the final

results were obtained. But actual experience had proved that the crystals should not be too large, and further, the presence of small crystals which could be regarded as embedded in the  $\beta$  solid solution was advantageous. Such an alloy would resist erosion almost as well as a pure  $\beta$  alloy and at the same time had the advantages of an  $\alpha$  alloy, which was able to stand fatigue better. Moreover, this alloy of mixed structure could be handled much better in the foundry. Propellers in use get knocked against dock gates and bawlers, and thus not infrequently suffered severe damage, to repair which was much more troublesome in a pure  $\beta$  alloy than in the case of an alloy of mixed  $\alpha\beta$  structure.

Another difficulty which took a good deal of overcoming was that of determining in what proportions the various ingredients for any given alloy should be taken in order to produce the desired composition when making comparatively large castings; for in operations of the type here dealt with it was no uncommon thing to have to run 20–30 tons of bronze at once, and when it was realised that alloys of this type alter in composition through loss of zinc, oxidation of manganese, and elimination of aluminium the whole time that they were in the reverberatory furnace and whenever they came into contact with air it would be seen that it was not easy so to adjust things as to arrive at an alloy varying not more than 0.1% or at most 0.2% in zinc, or an equivalent quantity of one of the substituting metals, which in the case of aluminium meant working to within ¼th of 0.2%. But these difficulties, like most others, had given way before persistence. The problem had started by being apparently a fairly simple one, because he had expected merely to find chemical corrosion, but, as the paper showed, this was far from being the case, and the work had involved something like 12 years' research.

## Manchester Section.

Meeting held at the Grand Hotel, on November 3, 1916.\*

MR. J. H. ROSEASON IN THE CHAIR.

### THE PREPARATION AND PROPERTIES OF 1,3,5-TRINITROBENZENE.

BY L. GUY RADCLIFFE, M.Sc.TECH., AND ALAN A. POLLITT, M.Sc.TECH.

The preparation of trinitrobenzenes presents interesting features in the light of the use of 1,3,5-trinitrobenzene as an explosive, but the literature relating to this subject is rather meagre, and for this reason the following research was undertaken.

Over a year ago experiments were made by one of us having for their object the determination of the conditions under which 1,3-dinitrobenzene could be further nitrated to form the trinitro derivative. In general, successful nitrations resulted in the production of the well-known 1,3,5-isomer, m.p. 121° C., but occasionally a trinitrobenzene of m.p. 61° C. was obtained, and this was at first thought to be the 1,2,4-compound described by Hepp (Annalen, 215, 362) and Lohry de Bruyn (Rec. Trav. Chim., 9, 190), though this compound is stated to melt at 57.5°.

It was soon found that this result could not be repeated at will, and numerous carefully conducted

\* Publication of this paper was deferred at the request of the Ministry of Munitions.

experiments have so far entirely failed to establish the conditions under which the trinitrobenzene of m.p. 61° can be produced.

A study of the properties of this new trinitrobenzene has caused us to modify the opinion that it was 1,2,4-trinitrobenzene, inasmuch as it has been found possible to convert the compound melting at 61° into the ordinary symmetrical 1,3,5-trinitrobenzene, m.p. 121°, by several simple methods. The facts appear to favour the assumption that 1,3,5-trinitrobenzene can exist in two forms which differ widely in their melting points. Hepp (Annalen, 215, 345) obtained 1,3,5-trinitrobenzene of m.p. 121°C. by heating 40 g. of 1,3-dinitrobenzene with a mixture of 300 g. of crystallised, fuming sulphuric acid and 120 g. of highly concentrated nitric acid (the exact strength of each is not stated), at first for one day at 80° C., and then for a further period of two days at 120°. The product was isolated by pouring the mixture into cold water, collecting the precipitate and washing it, first with water and then with dilute sodium carbonate solution, and finally crystallising the product from alcohol. Darst made the same substance by a very similar method.

Claus Becker (Ber., 16, 1597) obtained 1,3,5-trinitrobenzene from 2,4,6-trinitrotoluene by heating it to 180° with fuming sulphuric acid, carbon dioxide being evolved during the process. Furthermore, the same trinitrotoluene can be oxidised to trinitrobenzoic acid, and this latter when heated with water yields trinitrobenzene.

#### EXPERIMENTAL.

##### *Symmetrical or 1,3,5-trinitrobenzene.*

In the earlier attempts to prepare this substance mixtures of fuming nitric acid and fuming sulphuric acid (up to 60% SO<sub>3</sub>) were used, but even with a large excess of nitric acid and prolonged times of heating the dinitrobenzene was not nitrated. Further experiments were made with 100% nitric acid prepared by distilling a mixture of the 95% acid with 26% oleum. In the first of these nitrations 25 g. of dinitrobenzene was dissolved in 50 g. of oleum (26% SO<sub>3</sub>) and 20 g. of nitric acid (approximately 1½ mols. excess) added in small quantities at a time, the flask being kept cool during the mixing. The mixture was then gently heated for three hours at 110° C. in a wide-necked flask over a small flame and appeared to boil gently. The product was poured into water, washed, and crystallised once from alcohol; it formed plates or leaflets, m.p. 61° C., which proved on analysis (*cf. infra*) to contain 99.8% of trinitrobenzene. A 30% yield of the new compound was obtained. On repeating this experiment, using larger quantities, the product melted at 121° C., and was the ordinary symmetrical trinitrobenzene. Another experiment was made in which the quantities were 50 g. of dinitrobenzene, 75 g. of nitric acid (100%), and 125 g. of oleum (26% SO<sub>3</sub>). The two acids were mixed and heated to 110° C. and the powdered dinitrobenzene added gradually over a period of 1½ hours, after which the mixture was heated for a further 6½ hours at 110°–120° C. The product was isolated as usual, and on crystallising from alcohol plates of the *s*-trinitrobenzene, m.p. 121° C., were obtained; analysis indicated 100.2% of trinitrobenzene, the yield being 45% of the theoretical. In an attempt to increase the yield the same quantities as in the last experiment were used, and up to the completion of the addition of the dinitrobenzene the method of procedure was the same. At this point, however, a further quantity of mixed acids was added, consisting of 50 g. of oleum (26% SO<sub>3</sub>) and 35 g. of the nitric acid; the mixture was maintained at 110°–120° C. for 6½ hours, as in the last experiment. A 56% yield of trinitrobenzene, m.p. 121°, resulted, its purity corresponding to 99.97% trinitrobenzene.

A repetition of this method, using oleum containing 60% of SO<sub>3</sub>, in place of the 26% oleum, gave a yield of 60% of the usual *s*-trinitrobenzene; moreover, the time required was reduced to a total of 6 hours. In these experiments it was observed that a considerable quantity of the nitric acid was being lost by escaping from the flask, and therefore further nitrations were conducted under a reflux condenser, with the result that the loss of nitric acid was to a large extent prevented and the yield of *s*-trinitrobenzene slightly increased, viz., to 63%. Another series of experiments was made in which the reflux condenser was used, and, in addition, the reacting mixture was mechanically stirred throughout the experiment. In the first stage 50 g. of dinitrobenzene, 75 g. of nitric acid (100%), and 125 g. of oleum (60% SO<sub>3</sub>) were used, followed by 50 g. of oleum (60% SO<sub>3</sub>) and 35 g. of nitric acid (100%); working under these conditions a yield of 71% of *s*-trinitrobenzene, m.p. 121°, was obtained.

It has not been found possible to improve on this yield, as the use of higher temperatures increases the risk of the reaction getting beyond control, and if the time of the nitration is increased beyond about eight hours there is a reduction in the yield due apparently to the oxidation of the nitro compound.

At this stage it will be convenient to describe the nitrations which yielded a trinitrobenzene of m.p. 61°. In one experiment 25 g. of dinitrobenzene was dissolved in 50 g. of 26% oleum, 20 g. of 100% nitric acid was slowly added, and the resulting mixture heated for three hours at 110°, the product isolated melted at 61°, and analysis showed it to be a pure trinitrobenzene. The experiment was repeated several times, in detail, but on each occasion the resulting compound was pure *s*-trinitrobenzene, m.p. 121°, and numerous other nitrations failed to give the compound of m.p. 61°.

In an attempt to effect an economy in the amount of nitric acid used in the above method 45 g. of dinitrobenzene was dissolved in 125 g. of oleum (26% SO<sub>3</sub>), 20 g. of nitric acid (100%) added, and the mixture heated for three hours at 110°. On isolating the product, 26% of trinitrobenzene, m.p. 61°, purity 99.89%, was obtained, but again the repetition of the experiment gave the compound melting at 121°. These methods were frequently repeated and only very occasionally gave a product melting at 61°.

The failure to reproduce the experiment giving the compound melting at 61° seemed to indicate that some accidental trace of a catalyst might be causing its occasional formation, and therefore the effects of certain possible catalysts were tried, using the same conditions and quantities as in the two experiments just described.

First, a small quantity (0.5 g.) of flowers of sulphur was added to the nitration mixture, but beyond giving a slightly better yield of the ordinary *s*-trinitrobenzene it had no effect. Additions of ferric oxide, manganese dioxide, and urea produced no visible effect, the product being always the compound melting at 121°. It seemed possible that the presence of a trace of a reduced nitro compound, such as a nitraniline, might influence the nitration; to test this dinitrobenzene was mixed with a small quantity of sodium bisulphite before dissolving in the oleum, but this had no effect.

Whilst discussing the effect of catalysts the following interesting observation was made with regard to the effect of light on nitration. At a certain period it was found that the yields were not so good as usual. It was thought that as the windows of the laboratory had been darkened, at the request of the military authorities, by being covered with a blue wash, this subdued light might be the cause of the diminished yields, and what further nitrations were carried out in front of the

open window and in direct sunlight the original yields were obtained.

Another series of attempts was made to prepare the trinitrobenzene melting at 61° by using different strengths of oleum ranging from 20 to 60% SO<sub>3</sub>, increasing the strength each time by 10% SO<sub>3</sub>, the quantities being 25 g. of dinitrobenzene, 20 g. of nitric acid (100%), and 100 g. of oleum. In each experiment the nitric acid was added to the solution of dinitrobenzene in oleum and the mixture heated under a reflux condenser for three hours at 110°–120° C., with continuous mechanical stirring. The experiments gave without exception the variety melting at 121°, and the yields, though increasing with the strength of the oleum, varied very slightly. All manner of variations of these methods were tried, but in no case was the compound of m.p. 61° C. obtained. With the higher strengths of oleum it is not safe to exceed 120° C., especially when using a reflux condenser, as control over the reaction is very easily lost, but when using 20% or 30% oleum a temperature of 150° C. may be safely employed; furthermore, increasing the time beyond a certain limit causes loss of material and diminution in the yields.

It was now decided to alter still further the conditions of nitration, and with this object ordinary concentrated sulphuric acid (96% H<sub>2</sub>SO<sub>4</sub>) was used and sufficient phosphoric anhydride to take up the water in the sulphuric acid and also that produced during the nitration, with an additional 10% in excess of this theoretical amount: 37 g. of phosphoric anhydride was dissolved in 50 g. of sulphuric acid and 20 g. of dinitrobenzene dissolved in this mixture; the resulting solution was heated to 110° in an open flask and 12 g. of 100% nitric acid added from a tap funnel. After the addition of all the nitric acid the contents of the flask were heated for 3 hrs., attaining a maximum temperature of 150° C. after 1½ hrs., and maintaining this for the remainder of the time; no nitration occurred. A further 8 g. of nitric acid was added and the mixture again heated at 150° for a further 2 hrs.; a yield of 25% of *s*-trinitrobenzene, m.p. 121°, was obtained.

The experiment was repeated, starting with 20 g. of nitric acid instead of the 12 g. used at first in the previous attempt; the acid was added in three instalments of about equal volumes, the mixture being heated for 1 hr. to 130° after the first addition, for 1 hr. to 135° after the second, and for 3 hrs. to 150° C. after the third. This temperature was maintained for the remainder of the nitration. Although this method differs only slightly from the previous one, yet on isolating the product a yield of about 30% of the trinitrobenzene of m.p. 61° was obtained; analysis showed it to have a purity of 100.3%. During the nitration no oily layer formed, which solidified on cooling. It contained all the trinitrobenzene admixed with a very little unchanged dinitrobenzene, whilst the solid isolated from the residual mixed acid consisted almost entirely of unchanged dinitrobenzene. The formation of this oily layer has been observed in all the experiments in which phosphoric anhydride was used, no matter whether the compound produced melted at 121° or 61° C. This experiment was repeated exactly as detailed above, and again a 30% yield of the trinitrobenzene of m.p. 61° was obtained. Further repetitions, however, gave the compound of m.p. 121° C., and it has not been found possible to obtain further quantities of the compound of m.p. 61° C. by this method. No reason is advanced in explanation of these failures.

For the analysis of the products Knecht and Hibbert's method (see "New Reduction Methods in Volumetric Analysis," Longmans) was used, the trinitrobenzenes being dissolved in alcohol, reduced with titanous chloride solution, and the excess of the latter estimated by titration with iron alum.

The two varieties of trinitrobenzene are very similar in colour, being almost white, and both crystallise from alcohol in plates or leaflets made up of needles. Both forms are much less soluble than *m*-dinitrobenzene in alcohol; on crystallising the product isolated from a nitration, the first crop of crystals is almost pure trinitrobenzene, and by removing the second crop as soon as needles of dinitrobenzene begin to appear, a product is obtained which when once recrystallised is pure trinitrobenzene. This method of separation and purification is still easier if carbon tetrachloride is used instead of alcohol; the trinitrobenzenes are difficultly soluble in the former, whilst dinitrobenzene is comparatively very soluble. The trinitrobenzenes are easily soluble in ether and acetone and very soluble in benzene; both varieties are very slightly soluble in hot water and insoluble in cold water. The trinitrobenzenes dissolve in ammonia and caustic alkalis giving a deep red coloration, which is discharged by acids; they are not immediately affected by sodium carbonate or bicarbonate, but react slowly, producing a deep wine-coloured solution. The modification melting at 61° reacts with sodium bicarbonate in alcoholic solution much more slowly than does the variety melting at 121°. The colour is due to the formation of salts. By careful heating the ordinary form, m.p. 121°, may be sublimed, but it has not been found possible to sublime the variety of m.p. 61°.

With a view of establishing the constitution of the compound melting at 61°, the addition compounds of the two trinitrobenzenes with anilins were prepared. The molecular compound formed by adding aniline to the 1,2,4-trinitrobenzene, of m.p. 57.5°, is a red crystalline substance, m.p. 84°, whilst that obtained from 1,3,5-trinitrobenzene, of m.p. 121°, is also in the form of light red crystals of m.p. 124°. The aniline compound of the two trinitrobenzenes melting at 121° and 61°, as prepared by us, was made by adding aniline to each of the compounds and recrystallising the products from benzene; in both cases light red crystals were obtained, and from each variety the compound had the same melting point, viz., 124°. This remarkable result, wherein two compounds of widely different melting points gave an addition product with aniline having exactly the same melting point, dispersed the idea that the compound melting at 61° was 1,2,4-trinitrobenzene. On regenerating the trinitrobenzenes from the aniline-addition compounds by means of dilute hydrochloric acid each compound gave the same variety of trinitrobenzene, m.p. 121°. In this way the remarkable fact, that the trinitrobenzene melting at 61° could be converted into the well-known variety melting at 121°, was established. J. J. Sudborough (J. Chem. Soc., 1901, 522) has described the addition compounds of *s*-trinitrobenzene (m.p. 121°) with  $\alpha$ - and  $\beta$ -naphthylamine and their resolution by means of acetyl chloride. We have prepared the naphthylamine compounds of the trinitrobenzenes of m.p. 121° and 61° C., and find that both these varieties give a compound with  $\alpha$ -naphthylamine having m.p. 214° and with  $\beta$ -naphthylamine, m.p. 162°—the same figures as given by Sudborough. The trinitrobenzenes obtained by decomposition of these addition compounds melted at 121° C. in each case. These remarkable results lead to the conclusion that two forms of *s*-trinitrobenzene exist, and that the less stable form melts at 61° and is converted by simple reactions into the stable form, m.p. 121° C.

The conversion of the less stable into the more stable form was not accomplished by adding a crystal of the ordinary *s*-trinitrobenzene to a saturated alcoholic solution of the compound of m.p. 61°, but when equal volumes of the saturated solutions in alcohol of the two varieties were mixed and then fractionally crystallised only a compound of m.p. 121°–122° could be obtained. Heating the com-

pound of m.p.  $61^{\circ}$  for some time just above its melting point, either alone or in strong sulphuric acid solution, failed to bring about the conversion into the ordinary variety.

The molecular weights as determined by the Landsberger-Walker method, using acetone as solvent, were identical for both varieties, corresponding to a trinitrobenzene. The two varieties of trinitrobenzene were separately reduced with the calculated amount of strong titanous chloride solution and excess of hydrochloric acid in quantities sufficient to give a dinitroaniline from each of them; each variety yielded the same dinitro-aniline, in the form of bright yellow needles, m.p.  $159^{\circ}$ . Bader (Ber., 24, 1854) obtained 3,5-dinitro-anilins as yellow needles, m.p.  $159^{\circ}$ , by reducing 1,3,5-trinitrobenzene with ammonium sulphide; we have also reduced the two varieties of *s*-trinitrobenzene by the same method and obtained 3,5-dinitro-aniline, but the yield is not so good as that obtained by reduction with titanous chloride.

The nitrophenylenediamines and the triaminobenzenes were also prepared from each variety of the trinitrobenzene; the products appeared to be identical in each case.

From the above experiments there appears to be little doubt that *s*-trinitrobenzene can exist in two forms, and that one form is less stable than the other; it has not been found possible to control the conditions under which the less stable form is occasionally produced, but further investigations are in progress, and it is hoped that these points will soon be cleared up. In conclusion we desire to express our thanks to Mr. S. Medofski, B.Sc., Tech., for his assistance in some of the later parts of this work.

#### ADDENDUM.

Since reading the above paper the behaviour of trinitrobenzene melting at  $61^{\circ}$  and  $121^{\circ}$ , respectively, has been determined according to the method suggested by N. V. Sidgwick (Chem. Soc. Trans., 1915, 107, 672), and we are indebted to S. Medofski, B.Sc., Tech., for carrying out the experiments recorded below. Benzene was used as the solvent and its freezing point was determined, then the trinitrobenzene melting at  $61^{\circ}$  was added until the freezing point, which at first fell steadily, was constant. The trinitrobenzene melting at  $121^{\circ}$  was then added and the freezing point again determined. The depression of freezing point by trinitrobenzene of m.p.  $61^{\circ}$  was  $2.474^{\circ}$  (solubility 85.0 g. per litre), and the depression by the mixture of trinitrobenzenes, m.p.  $61^{\circ}$  and m.p.  $121^{\circ}$ , was  $2.468^{\circ}$ . It is evident, therefore, that the concentration of the solution has not increased by the addition of the compound melting at  $121^{\circ}$ , and that, according to Sidgwick, the forms are polymorphic.

School of Technology,  
Manchester.

## Communication.

### THE RELATIVE ACTIVITY OF VARIOUS ALLOTROPIC FORMS OF SULPHUR TOWARDS CAOUTCHOUC.

BY D. F. TWISS AND F. THOMAS.

The investigations in recent years of the allotropic forms of sulphur capable of existence within the range of temperature used in ordinary vulcanisation, naturally prompt enquiries as to a possible difference in the activity of these various modifications of the vulcanising agent. The forms most likely to be involved are (a)  $S_A$ , the modification

represented by ordinary crystalline sulphur, (b)  $S_B$ , the insoluble modification constituting part of gennine "flowers of sulphur" and corresponding with the viscous constituent of molten sulphur, and (c)  $S_r$ , a more brightly coloured and more soluble variety, present in a small proportion with much  $S_A$  in the equilibrium mixture yielded by ordinary sulphur at temperatures a little above its melting point; the proportion of  $S_r$  increases markedly if the temperature is raised to  $140^{\circ}$  or higher (Aten, Z. physik. Chem., 1912, 81, 257; 1913, 83, 442; 86, 1. Proc. K. Akad. Wetensch., Amsterdam, 1913, 20, 824).

Tests made with mixtures of rubber with soluble sulphur ( $S_A$ ) and insoluble sulphur ( $S_B$ ) respectively, under ordinary technical conditions, indicate that the vulcanising effect of these two varieties is practically the same (Twiss, Annual Reports of the Progress of Applied Chemistry, 1919, 4, 327); this result, however, is probably to be attributed to the rapid change of  $S_B$  at such temperatures into the equilibrium mixture consisting mainly of  $S_A$  with some  $S_r$  (Beckmann and others, Z. anorg. Chem., 1918, 102, 201; 103, 189).

Between  $128^{\circ}$  and  $168^{\circ}$  C. the temperature coefficient of the chemical reaction between rubber and sulphur, i.e. the proportion by which the rate is increased on raising the temperature  $10^{\circ}$  C., is surprisingly constant, and in view of the alteration of the composition of the equilibrium mixture with rise of temperature, the comment has been made that this is indicative of comparable vulcanising capacity on the part of the modifications present (Twiss and Brazier, J., 1920, 125 r).

Although the difference observable between the forms of sulphur naturally existent at ordinary vulcanising temperatures is thus shown to be relatively slight, it must be remembered that under such conditions the sulphur will undergo fusion and then rapidly yield the equilibrium mixture. If the temperature could be maintained much lower, the rate of attainment of equilibrium could be reduced and the increased persistence of each form would afford greater opportunity for observing any difference in vulcanising capacity. The use of a suitable vulcanisation catalyst, e.g., aldehyde-ammonia, enables experiments to be made at a sufficiently low temperature. It is a little unfortunate that the powerful organic catalysts generally should be basic substances such as also tend to catalyse the mutual inter-conversion of the sulphur allotropes, but, as is demonstrated by the results now submitted, the disturbance from this direction is not sufficient to mask completely the sought effect.

In the first set of experiments the varieties of sulphur used were (a) a high grade finely powdered sulphur soluble in carbon bisulphide and consisting entirely of  $S_A$ , and (b) an extracted sublimed flowers of sulphur 93% insoluble in carbon bisulphide and consisting mainly of  $S_B$ . Mixtures were made of each of these with selected pale crepe rubber of known rate of vulcanisation; the composition in each case was rubber 90 parts, sulphur 10 parts, and aldehyde-ammonia 1 part. In order to ensure as closely comparable composition and conditions as possible, the sulphur in both cases was first mixed with an equal weight of the rubber and the composition of each stock checked before introducing the correct amount of each into the final mixtures. Similarly the aldehyde-ammonia was first mixed with 9 times its weight of the rubber so as to increase the accuracy with which equal proportions of this "stock" could be introduced into the two batches of equally "worked" rubber. Vulcanisation was effected, with the two mixtures simultaneously, at  $98^{\circ}$ ,  $108^{\circ}$ , and  $118^{\circ}$  C. Figure 1 shows the rate of vulcanisation as indicated by the gradual reduction in extensibility of the two mixtures at the respective temperatures; the trans-



verse dotted lines mark the approximate position of the "optimum tensile strength" where this was observed. Figures for combined or residual free sulphur are not given because their satisfactory determination in the ordinary way is impossible in the presence of  $S_{\mu}$  (see Twiss, J., 1917, 789).

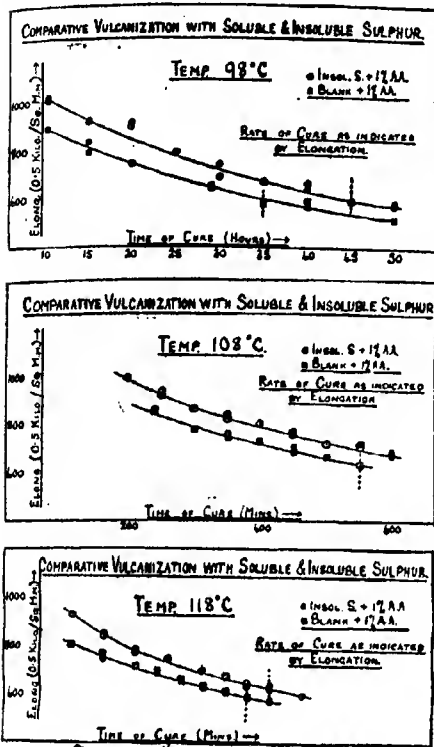


FIG. 1.

It will be noted that under the conditions of these experiments the vulcanising effect of  $S_{\mu}$ , particularly at the lowest temperature, is definitely inferior to that of SA.

Taking an extensibility of 700% as a standard for comparison, the difference in the rate of vulcanisation of the two mixtures is clearly seen from the respective periods required, and the more rapid conversion of  $S_{\mu}$  into SA with rise of temperature is reflected in the corresponding decrease in the temperature coefficient.\*

Rate of vulcanisation.					
$S_{\mu}$ (93%).			SA.		
Time required.	Temp. coefft.		Time required.	Temp. coefft.	Relative effect, $S_{\mu}$ : SA.
98° C.	32 hours	—	23 hours	—	1 : 1.4
108°	550 mins.	8.5	440 mins.	8.1	1 : 1.3
118°	185 mins.	8.0	140 mins.	8.1	1 : 1.3

\* In these experiments vulcanisation was effected uninterruptedly for the full periods, the temperature of the oil bath being maintained constant for the whole of the time. The greater value of the temperature coefficient obtained previously with SA for the interval 98°-108° C. (Twiss and Breaker, loc. cit.) is to be ascribed to the fact that the earlier experiment at 98° C. was made in two stages, with the result that the vulcanisation would be retarded somewhat on account of the partial separation of sulphur from solution in the rubber half-way through the experiment.

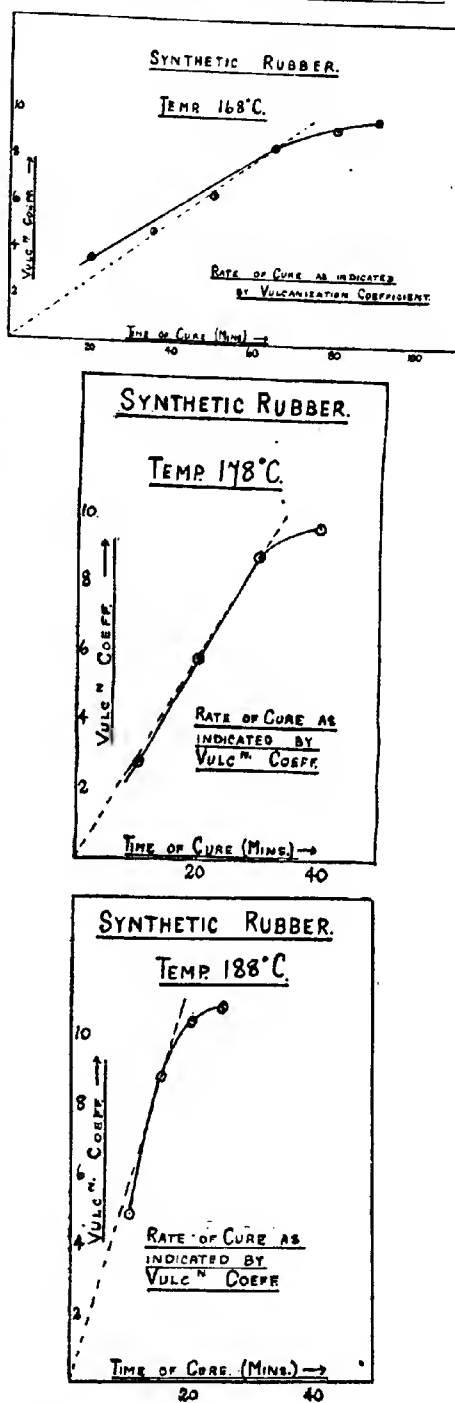


FIG. 2

An experiment with similar mixings of the same rubber and two forms of sulphur in the same proportions, but without the catalyst and at 148°, confirmed the earlier observations (Twiss, loc. cit.) as to the closely comparable effectiveness of the  $S_{\mu}$

and  $S_\lambda$  at this temperature, the relative effectiveness calculated in the same way as above being 1:1.06.

These results as a whole demonstrate that at the ordinary vulcanising temperatures, *e.g.*,  $148^\circ$ ,  $S_\mu$  changes so rapidly into  $S_\lambda$  that no appreciable difference can be detected in the rate of vulcanisation, the effect in each case being that actually produced by  $S_\lambda$ . At lower temperatures it is possible to detect a distinctly feeble effect in the mixture containing  $S_\mu$ . The possibility that this difference may be characteristic not of  $S_\mu$  but of  $S_\pi$ , which is formed together with  $S_\lambda$  from the less stable  $S_\mu$  at such temperatures, is hardly in accord with the almost identical effect observed at  $148^\circ$  C. (see above) and with the results given below. In all likelihood therefore the difference arises from the  $S_\mu$  itself and is probably not explained completely by the lower solubility of this form of sulphur.

From the constancy of the temperature coefficient of the reaction between ordinary sulphur and rubber at  $138^\circ$ – $168^\circ$  C., over which range  $S_\lambda$  gives rise to an increasing proportion of  $S_\pi$ , it would appear that  $S_\pi$  and  $S_\lambda$  are of almost equal activity in this direction. This result is of interest in view of Aten's observation (*Z. physik. Chem.*, 1914, 88, 321) that  $S_\pi$  retards the action of  $S_\lambda$  on silver and mercury.

There appeared to be some hope of obtaining more evidence of the activity of  $S_\mu$  relative to  $S_\lambda$  by making vulcanisation experiments with rubber at higher temperatures, at which sulphur is known to undergo a marked change in molecular complexity probably corresponding with a rapidly increasing proportion of  $S_\mu$  in the equilibrium mixture (Beckmann and others, *loc. cit.* Kellas, *Trans. Chem. Soc.*, 1918, 113, 903. Smith, *Proc. Roy. Soc. Edin.*, 1905, 25, 588, 590; Smith and Carson, *ibid.*, 1906, 26, 352). Unfortunately with ordinary rubber the chemical change accompanying vulcanisation would then be too rapid for convenient examination, and recourse was therefore had to the use of synthetic rubber which, as is well known, is relatively sluggish in vulcanisation; the sample used was of "methyl-rubber," *i.e.*, polymerised dimethylbutadiene, which was free from artificial catalysts and "elastomers." On account of the hardness of the vulcanised products obtained with such synthetic rubber, no physical tests were possible and the rate of reaction was followed only by the gradual disappearance of sulphur into combination. The composition of the original mixture was sulphur ( $S_\lambda$ ) 10.4%, methyl-rubber 89.6%. Experiments were made at  $168^\circ$ ,  $178^\circ$ , and  $188^\circ$  C., the results being as given in figure 2.

It is remarkable that the disappearance of most of the sulphur into combination appears to follow the approximately rectilinear course already observed with natural rubber (Skellon, *Rubber Industry*, 1914, 172; van Iterson, *Comm. Netherland Gov. Inst. for Advising Rubber Trade*, 1918, 7, 247); if, as suggested by van Iterson, this is due to autocatalysis it is evident that the non-caoutchouc constituents of natural rubber are not responsible.

The results are not quite as smooth as could be desired, but if comparison is made of the time required for the attainment of the various degrees of vulcanisation at different temperatures, the temperature coefficients in each case are greater for the interval  $168^\circ$ – $178^\circ$  than for  $178^\circ$ – $188^\circ$ .

The apparent slight increase in the temperature coefficient during the progress of the reaction

appears to indicate the existence of a very brief initial period of greater reactivity. This may be due to the fact that the sulphur initially is composed practically entirely of  $S_\lambda$ , which rapidly undergoes conversion into the less active equilibrium mixture; it might, however, be caused by the presence in the synthetic rubber of a small proportion of some more active material. There is no such regular alteration observable in the figures calculated in a similar way from the experiments with ordinary rubber and sulphur at  $138^\circ$ – $168^\circ$  C. (Twiss and Brazier, *loc. cit.*).

A better comparison of the temperature coefficients is probably given by the ratio of the tangents of the angles included between the horizontal axis and the line (dotted in Fig. 2) between the origin and the point representing the end of the "rectilinear" course of the vulcanisation process. This method of comparison likewise shows a smaller increase in the rate of reaction for the second interval.

Vulcan. coefft.	Time (mins.).			Temp. coefft.	
	$168^\circ$ .	$178^\circ$ .	$188^\circ$ .	$178^\circ$ – $168^\circ$ .	$188^\circ$ – $178^\circ$ .
4	25	14	—	1.8	—
5	34	18	10	1.9	1.3
6	43	21	11	2.0	1.4
7	52	24	12	2.2	2.0
8	61	28	14	2.2	2.0
9	73	31	15	2.4	2.1
angle	$33^\circ$	$56^\circ$	$71^\circ$	—	—
tan. angle	0.65	1.43	2.90	2.3	2.0

The smaller coefficient for the higher temperature interval suggests that the sulphur equilibrium mixture at the higher temperature is relatively less effective than the equilibrium mixture existent at the lower temperature; as the difference in the composition of the two equilibrium mixtures will be a smaller proportion of  $S_\lambda$  and a greater proportion of  $S_\pi$  and  $S_\mu$  at the higher temperature, and as the earlier considerations lead to the view that the effectiveness of  $S_\pi$  is very little different from that of  $S_\lambda$ , it follows that the results again indicate a somewhat inferior vulcanising capacity for  $S_\mu$ . It must be remembered, however, that the observed differences in such experiments, based on the relative activity of interchangeable modifications of a chemical substance, will be diminished by the tendency of the equilibrium automatically to adjust itself as the more active form disappears.

Although these results indicate a difference between the activity of  $S_\mu$  and of  $S_\lambda$  or  $S_\pi$ , the outstanding fact is not the existence of this difference but its relatively small magnitude. The three forms are of such diverse general characteristics that much greater differences might have been expected. Under the conditions of ordinary technical practice indeed, the possibility of alteration in the relative proportions of the various modifications of sulphur is not likely to form an appreciable disturbing factor in vulcanisation, and the only instance of any special form of sulphur (as such) showing special vulcanisation features appears to be that possibly involved in the recently discovered method of treating raw rubber with sulphur dioxide and hydrogen sulphide.

For the experiments recorded above, use was made of the very convenient oil-bath method of vulcanisation.

## London Section.

Meeting held at Burlington House on January 3, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

### THE SMITH CONTINUOUS SYSTEM OF CARBONISATION.

BY GEORGE H. THURSTON, M.I.E.E., A.M.S.M.E.

The carbonisation of coal has been fully dealt with in this country but up to the present, so far as the author is aware, no complete description has been given of the system designed and perfected by Mr. Charles H. Smith, of New York, or of the plants constructed on this system and producing a smokeless fuel called "Carbocoal" at the rate of 550 tons per day. One of these plants situated at Irvington, New Jersey, has been in constant operation for the past two years.

The Smith continuous system is a combination of low- and high-temperature carbonisation, and the inventor has retained all the advantages connected with the high oil recovery in the former, and in addition has succeeded in increasing the yields of tar and sulphate of ammonia by means of the latter, at the same time producing a free-burning smokeless fuel that stands rough handling with a minimum of loss.

In order to obtain low operating costs on a carbonisation plant it is essential that the process should be continuous, and lost time reduced to a minimum. Full advantage must be taken of labour-saving machinery and manual labour eliminated as much as possible. To carry out this effectively necessitates the construction of a plant capable of dealing with at least 200,000 tons of coal per annum. The importance of these features is fully realised in the United States, and in the 150,000-ton carbocoal unit of the 1,500,000-ton plant built at Clinchfield, Va., for the International Coal Products Corporation of Virginia, to operate the Smith process, labour-saving devices are used to the utmost extent. This plant commenced producing in July, 1920, and is working smoothly.

An objection frequently raised to the fuel obtained from low-temperature carbonisation is its friability and bulk, which make its transport difficult and costly. "Carbocoal" is carried on the railways in the United States at the same rates as coal, and is not subject to these disadvantages.

In the Smith continuous system of carbonisation the coal, which is crushed to pass a  $\frac{1}{2}$  in.-mesh screen, is subjected to a primary carbonisation at a temperature of about 480° C. to reduce its volatile content to a uniformly low figure, whereby it is converted into a friable residue containing 7–10% of volatile matter, called "semi-carbocoal." The period of this process is 2–3 hours, and the operation is continuous.

After passing through the retort, the material is ground and mixed with a suitable percentage of pitch and briquetted. The briquettes, which contain about 15% of volatile matter, are then heated to 1000°–1100° C., which drives off all but 3 or 4% of the volatile content and leaves them firm and hard. The quality of the briquettes is influenced by the ash content of the coal, but since this is evenly distributed throughout the mass, the clinkering of the fuel during combustion is absent except when the ash has a very low fusion point, and it has been found that, although the coal loses 2% of its calorific value by carbonisation, the actual heating efficiency of Carbocoal—referred to the coal from which it is made—is 94%.

The tar recovered from the primary retort and by scrubbing the gas contains the usual oils and ammonium sulphate found in tar recovered by the low-temperature process. The yield, owing to the low temperature employed, is high, the actual quantity recovered and its value naturally depending on the constituents of the coal. The tar recovered from the secondary carbonisation contains a large percentage of heavy oil and pitch, and when distilled considerably increases the total yield of ammonium sulphate.

The gas produced in the Smith system varies in quality and quantity; that from the primary retort has a calorific value of 650–700 B.Th.U. per cb. ft., and the quantity distilled from a 35% volatile coal is between 5600 and 6700 cb. ft. per ton. In the secondary retort the gas has a value of 350–400 B.Th.U. and the yield is about 4480 cb. ft., or an average total of 10,630 cb. ft., with a calorific value of about 543 B.Th.U. In the present stage of development, the system, after supplying all the demands, furnishes a surplus of 2240 cb. ft. of gas.

#### Description of plant.

By referring to Fig. 1, which illustrates a typical flow diagram of a plant laid out to produce "Carbocoal" by the Smith continuous system of carbonisation, it will be seen that the coal is delivered into the track hopper or storage bin, *a*, and transported by means of the belt conveyor, *b*, to the hopper of the coal crusher, *c*, which reduces it to about  $\frac{1}{2}$  in. size; the fine coal is then raised by the elevator, *d*, and distributed by means of the conveyor belt, *e*, to the hoppers, *f*, which automatically feed the primary retorts, *g*, a detailed description of which will be given later (see also Figs. 2, 3, and 4). The black, friable, spongy mass formed in the primary retort is conveyed by means of the paddles in the retort to the discharge orifice, where it is deposited in pieces not over 6 in. in size in the water-jacketed discharge hopper, *h*, situated immediately below the retort. From this it is fed by the worm, *i*, to the conveyor, *j*, which transfers it to the storage bin, *k*.

The mass is then fed by means of a feeder conveyor, *l*, to the crusher, *m*, where it is broken up to  $\frac{1}{4}$  in. or less, after which it is raised by the elevator, *n*, to the mixer, *o*, in which it is mixed with 8–10% of its weight of liquid pitch, stored in the vessel, *r*. The mixture then goes to the "fluxer," where the two materials are intimately mixed in conjunction with steam which melts the pitch, and passes from there into a briquetting press, *q*, of the roll type. The raw briquettes are then passed to the storage bin, *u*, by means of a cooling conveyor, *t*. This conveyor is of sufficient length to enable the briquettes to be completely set by the time they arrive at the storage bin, and they are thus ready for the secondary carbonisation. Underneath the briquetting press is a second conveyor, *s*, for recovering the fines made from the press and returning them to the fluxer, so that only whole briquettes are carried to the storage bin. The briquettes are taken from the storage bin by means of a lorry car, *v*, and deposited in the secondary retort, *w*, where the final carbonisation is completed.

The high-temperature retort illustrated in Fig. 5 is a modification of the inclined gas retort; the modification consists of the compartments being made rectangular in place of the usual D shape; the retort has three compartments each containing two carbonising chambers that are charged and discharged by gravity.

The temperature to which the briquettes are subjected is approximately 1090° C., which expels practically all the volatile matter. The time occupied in this operation is about six hours per charge. The briquettes are then discharged by gravity into the

car, *x*, transferred to the loading station by means of the track, *y*, and quenched with water, and are then ready for despatch.

#### The Smith retort.

The retort invented by Mr. C. H. Smith constitutes the most important link in the carbonisation system developed by him. The retort, which is cylindrical and about 18 ft. long, has an inverted heart shape (see Fig. 2). It is constituted of individual blocks of carborundum containing a special asbestos-packed tongue-and-groove expansion joint. Carborundum was selected after long and extended trials, in which retorts constructed of other refractory material and metal were used. When its heat-conducting and wearing qualities are taken into consideration it is considered to be the best material for the purpose that has been discovered up to the present. The end walls of the retort are of cast iron lined with firebricks and contain the necessary openings for the feed and the paddle shafts, which are parallel with the axis of the retort. The shafts pass through asbestos-packed stuffing-boxes, forming part of the cast-iron covers on the end walls, and are carried by substantial sleeve bearings. The paddles for stirring the material and moving it through the retort have a radius of 2 ft. 3 in., and the clearance between the end of the paddles and the carborundum muffle blocks is  $\frac{1}{2}$  in. The paddles, which are bolted to the 12-in. shaft, are mounted alternately, so that each paddle wipes the opposite shaft, and its face is inclined sufficiently to push the material through the retort. The shaft makes one revolution in eight minutes, and the power required is 2 B.H.P. It has been found in practice that the speed can be considerably accelerated if it is considered desirable to stir up the coal to a greater extent. Under average operating conditions it is the practice to keep the retort about half full, which provides ample room for intumescence of the coal without interfering with its operation.

The feeding is accomplished by means of a single screw conveyor running in a closed pipe; the packing of the material is depended on for the necessary gas seal. The conveyor is driven by a motor, and the average power taken is about 5 B.H.P. The regulation of the feed is effected by starting and stopping the motor by means of contacts mounted on the paddle shaft gear wheel.

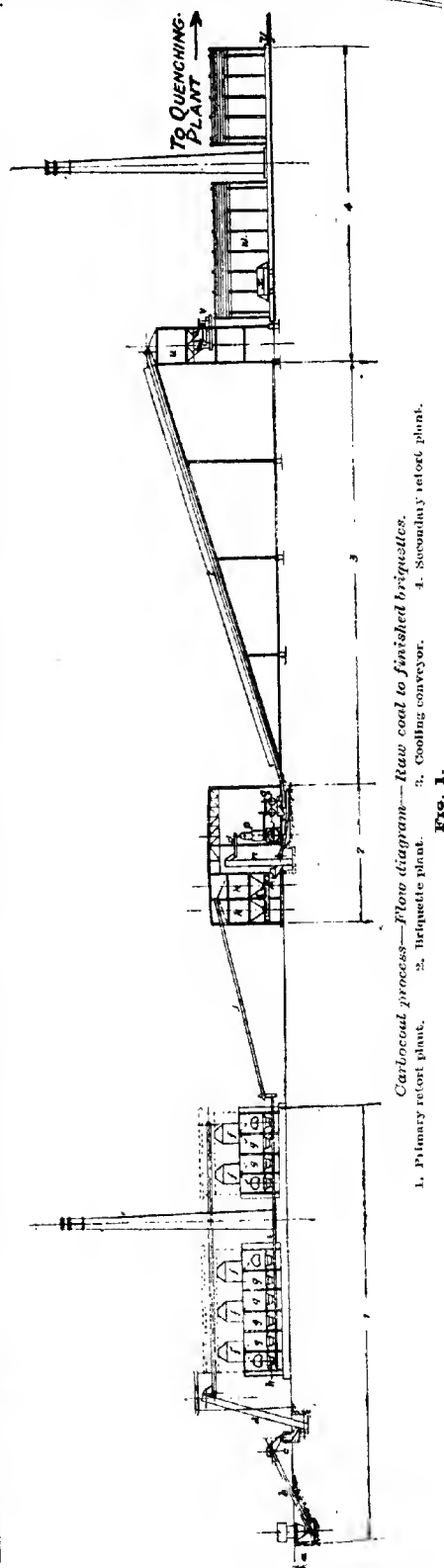
The apparatus for discharging the material is situated at the bottom of the retort, and consists of arms mounted on a revolving shaft, which serves to break up the product as received from the retort and deliver it in a suitable condition to a screw conveyor operating in a water-cooled pipe turned up at the end to form a gas seal.

Stripped gas is used for heating; the burners are placed in the flues of the brick setting surrounding the retort, which also contains the usual recuperative features for pre-heating the air by means of the heat in the waste gases. Sufficient heat is maintained in the retort so that the outgoing carbonised product has a temperature of about 480° C. When treating American coal containing 35% of volatile matter and 0.72% of moisture the retort has a capacity of one ton per hour.

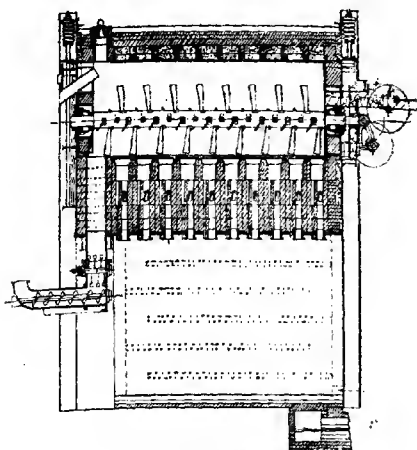
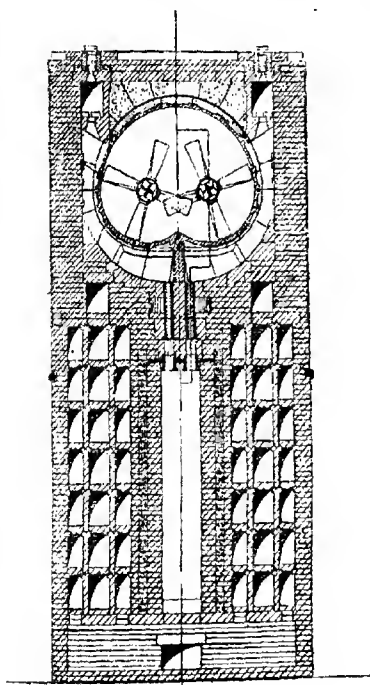
#### Products of distillation.

The products of distillation pass out at the top of the retort and are not subjected to any increase in temperature, so that the tar obtained possesses all the characteristics of a low-temperature tar, and the yield is exceptionally good, as will be seen from the following results obtained from a ton of typical high volatile Pittsburgh coal:—

	Briquettes.	Primary furnace.	Secondary furnace.	Total 70% dry.
Gas, cu. ft.	5600	4430	..	10,030
Tar (80% tar acids), galls.	22.4	5.6	..	28
Sulphate of ammonia, lb.	2.24	20	..	22.24



Carbonisation process—Flow diagram—Raw coal to finished briquettes.  
1. Primary retort plant. 2. Briquette plant. 3. Cooling conveyor. 4. Secondary retort plant.

Primary retort—longitudinal section.  
FIG. 2.Primary retort—cross-section.  
FIG. 3.

The 28 gallons of tar when distilled gives a yield of 18.6 gallons of distillate, the remainder being pitch, which is used in making the briquettes. If, on investigation, it is found profitable to "crack" the tar into motor spirit, naphtha, tar acid, and fuel oils, this can be accomplished by the installation of the usual by-product plant. It is estimated that all the necessary heat and power required for the by-product plant will be furnished by the 2240 cb. ft. of surplus gas obtained from the carbonising process. The by-product plant is well known, and as it does not form an integral part of the plant it is not the intention to devote any further space to its description.

INTERNATIONAL COAL PRODUCTS CORPORATION.  
TABLE I.—Selection of results from tests of 35 various coals and lignites.

Sample from mine.	Moisture as received, %	Proximate analysis (dry basis).										Yields total products per short ton of coal (dry basis).				
		Raw coal.					Semi-carbonized.					Carbo- nized lb.	Tar, galls.	Ammonia lb.	L. oil from gas, galls.	Gas, cb. ft.
		Vol.	Fixed C.	Ash.	B.Th.U.	Vol.	Fixed C.	Ash.	B.Th.U.	Vol.	Fixed C.	Ash.	B.Th.U.			
American coal—																
Marathon, Ky.	3.28	38.60	58.2	4.3	14,780	9.5	80.27	11.16	13,080	3.24	80.52	8.30	13,320	21.48	1.57	7,657 (583 E.Th.U.)
Marathon, Ky.	3.38	20.20	53.38	8.42	14,568	7.78	81.07	11.16	13,080	1.24	86.88	11.44	13,320	19.05	0.747	8,020
Marathon, Ky.	1.72	33.50	57.87	0.63	14,568	14.09	77.09	8.22	13,563	3.17	86.75	10.03	12,970	22.04	1.89	7,900 (512 E.Th.U.)
Marathon, Ky.	26.35	45.21	41.50	13.29	11,055	11.07	07.05	21.28	10,039	8.17	70.43	23.40	11,275	21.53	2.03	11,232 (555 " )
Walsen Washed, Nuts,	3.85	37.35	48.53	14.12	12,569	14.41	65.73	19.86	11,786	6.52	73.61	19.47	11,030	39.44	1.80	9,011 (552 " )
W. Ohio, (Ill. coking),	15.40	40.40	53.00	6.00	13,807	9.76	80.53	9.71	13,540	3.21	86.81	0.08	13,087	39.00	1.70	9,037 (546 " )
Rouse West, Colo. (non-	2.13	38.68	50.38	10.04	12,237	10.42	74.39	15.19	12,600	3.52	81.10	15.29	11,831	19.95	2.08	8,233 (528 " )
Canadian coal, (coking coal)	3.04	25.64	61.76	12.57	13,472	7.30	76.28	16.42	12,132	2.13	82.40	15.47	12,280	10.52	1.32	8,976 (416 " )
Taylor, Can. (lig)	35.00	45.20	42.30	12.50	11,006	14.72	67.23	18.63	11,782	3.54	79.65	16.81	11,370	24.70	2.07	13,432 (391 " )
Nova Scotia (coking coal)	1.53	32.10	56.46	11.44	13,432	8.60	75.75	15.35	12,180	3.50	80.24	16.20	11,405	18.50	1.56	8,816 (545 " )
Japanese coal—																
Ubeo, Hokkaido	19.00	39.70	42.12	13.18	11,142	15.92	62.65	21.43	10,680	3.80	71.15	25.05	9,330	23.04	1.11	7,840 (530 " )
Ubeo, Hokkaido	1.44	39.36	54.00	7.04	13,000	9.20	79.67	11.15	12,690	4.92	83.23	15.38	11,555	10.09	2.47	8,800 (502 " )
Takashima Inapp (coking)	3.21	37.50	43.72	18.78	11,408	10.46	62.68	20.96	9,448	3.50	67.55	28.95	9,798	13.06	2.84	9,614 (448 " )
Shimizu Lump (non-																
Ota road—	7.96	35.10	55.64	9.26	14,277	10.98	76.35	19.07	13,772	4.03	89.25	13.47	13,651	15.03	1.97	9,254 (554 " )
Maries, France	10.32	29.03	46.60	23.75	—	12.51	50.89	36.00	—	5.63	60.44	33.92	—	18.00	0.65	6,612 (525 " )

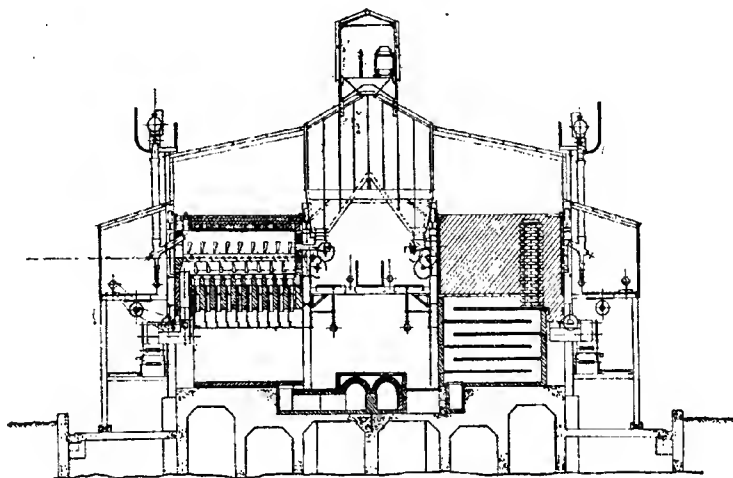
*Pre-heating.*

That the Smith retort is capable of dealing successfully with a great variety of fuels can be seen by referring to Table I., and there is no apparent reason why British fuel should present any greater difficulties than those already treated in the retort. It is obvious that the results obtained from the different kinds of fuel will vary, and that careful investigation is necessary before laying out a plant.

removed from the tar, the work performed in the more expensive unit is reduced to a minimum.

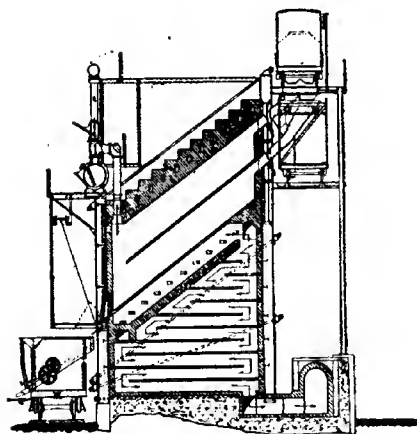
*Use of Carbocoal in boilers, furnaces, and blast-furnaces.*

Judging from the published reports of tests made by the United States Navy Department, carbocoal, when burnt in the furnace of a Babcock and Wilcox boiler, compared favourably with the coal from



Primary retorts—general cross-section.

FIG. 4.



Secondary retort—cross-section.

FIG. 5.

The investigation may point to the necessity of washing the coal, with the possibility of having to subject it to preliminary drying for the purpose of driving off the moisture before being fed into the primary retort. To accomplish this a simple form of dryer has been designed by Mr. Smith. In this device the waste gases from the retort are passed through it on the way to the chimney. By means of this the moist coal is heated to 150° C., and the period of distillation in the primary retort is reduced proportionately. In addition a considerable amount of moisture is eliminated from the gas by this pre-heating, and, as this does not have to be

which it was made. The tests indicated that one pound of Carbocoal evaporated 9.09 lb. of water from and at 212° F., as against 9.59 lb. of water for the coal it was derived from. Similar tests were also made on a horizontal tubular boiler at the Fosdick Machine Tool Company's works. The fuels used were bituminous slack, having a calorific value (dry) of 14,120 B.Th.U., as against 12,470 B.Th.U. for carbocoal. The relative evaporation was 6.69 lb. and 7.34 lb. of water per hour from and at 212° F.

Carbocoal has also been tried with success in a locomotive. One very valuable feature of the briquettes is their great strength, which has been obtained without sacrificing their porosity. This feature enables the fuel to be roughly handled and transported long distances with a minimum of loss due to breakage. Recent tests have demonstrated that it has all the advantages of coal as a powdered fuel and at the same time is immune from spontaneous combustion. This, in addition to its occupying very little more space than an equal weight of coal, makes it an excellent bunker fuel for marine work. The hardness of carbocoal and its great strength when subjected to heavy pressure make it suitable for use in the blast furnace; this is of considerable importance, as it indicates that a fuel suitable for metallurgical purposes can be produced by means of the Smith system from coal which the coke oven is incapable of dealing with.

Tests to ascertain the crushing strength of carbocoal made by Messrs. Kalmus, Comstock and Wescott, Inc., Industrial Research Engineers, of Boston, U.S.A., in conjunction with Dr. Walter Runge, of the International Coal Products Corporation, indicate that the average crushing strength was 943 lb. per square inch at a temperature of 1400° C.; the crushing strength of coke at room temperature was 944 lb. per square inch. The apparent specific gravity of both carbocoal and coke was found to be 0.973 at room temperature.



## DISCUSSION.

Dr. W. R. ORMANDY said that the plant described appeared to him to resemble the Del Monte retort with two stirrers instead of one, whilst the soft coke was made into high-temperature coke. As far as he could gather, in this process the gases given off by low-temperature distillation were added to those given off by high-temperature distillation, and a large amount of pitch was added to the coke. With the Del Monte process, in which a continuously moving worm was working in a sticky mass of good Durham gas coal, there would result a soft, friable coke, which would be very porous. If that coke were mixed with pitch and heated to  $1090^{\circ}\text{C}$ ., there was strong presumptive evidence that a hard coke would result, which would probably stand the pressure of the blast furnace. The description of the Smith process led him to think that an effort was being made to claim the advantages both of low- and high-temperature distillation. If the process gave a hard coke this would not have the advantages which were attached to low-temperature coke, which would ignite readily and was an admirable fuel for ordinary household use. The harder coke might be better for metallurgical purposes, and would probably be very suitable for steam raising, and it would certainly be better for transport purposes. On the whole, however, it did not seem to him that there was a great deal of novelty in this beyond the attempt which had been forecasted by many writers, to get over the difficulty of the soft, fine coke produced by a really low-temperature process by briquetting it, and then reheating the briquettes in this semi-continuous process. It was interesting to find that, although the primary process was to be carried out at a temperature not exceeding  $480^{\circ}\text{C}$ ., the inventor did not seem to have found that an iron retort was satisfactory, and had resorted to the use of carborundum. That material had been used years ago by Dressler for making the combustion tube of his continuous ceramic furnace; in this, however, it was used, with a similar sort of expansion joint, at a temperature of  $1500^{\circ}$ – $1700^{\circ}\text{C}$ ., where a metallic material could not be used. He failed to see what advantage there was in the use of a carborundum tube in the place of an iron tube in the present system. It seemed to him also that the Del Monte system of taking the gas off from a number of points from the top of the retort was an advantage compared with using the carborundum retort with the gas exhaust at one end, so that the products of distillation were exposed for a much longer time to the temperature of the inside of the hot retort.

Dr. R. LESSING said that the process appeared to be working on a fairly extensive scale. He believed the American Government had given a grant in connexion with the plant. He agreed with Dr. Ormandy that there might be features of the process which could not be claimed to be novel, but to-day that was really not the question in low-temperature carbonisation. It was rather a question of applying well-known principles in a workmanlike fashion, and, what was more important, in a manner which was based on scientific investigations, because what was missing to-day was the scientific data on which plants could be constructed. Until these data were obtained in a great mass for any number of coals, and, what was equally important, he ventured to think, in connexion with the materials of which retorts had to be made, the progress which was desired would not be made. We knew next to nothing to-day of the conductivity of coals under the conditions under which large masses had to be carbonised, and it was this question of conductivity that was responsible for all the failures that had been experienced hitherto. They had to deal with a problem in which a great deal of heat had to be forced into a material which was less conducting

than the coke or semi-coke obtained at high temperature, and this had to be done from a lower starting point in order not to decompose the material at the walls of the retort to the same extent as at high temperature. They had to deal, therefore, with a very much less steep heating gradient, and that was the crux of the failures hitherto experienced. Until these matters had been thoroughly thrashed out in a scientific manner there was very little hope of getting this problem really solved. On the other hand, there was not the slightest doubt that the problem was one worthy of solution, and any money spent in this direction, provided it was done on really scientific lines, ought to produce results of great economic value.

Mr. E. LAWSON LOMAX, referring to the quality of the oils produced by low-temperature carbonisation processes, said that all the advocates of these processes claimed to get so many gallons of oil per ton of coal carbonised. They did not, however, say what kind of oil it was nor whether it was free from water when measured. From his experience in handling these oils he had found that most of them were very difficult to refine, and also it was very difficult to get accurate readings of the quantities, owing to the fact that so much water was held in suspension. At the present time, and having regard to our present knowledge of the chemistry of these oils, they could not be classed as having any value except for fuel purposes, and therefore in most cases the values assigned to them by the advocates of the various processes were fictitious. The figure of 20 to 30 cents per gallon mentioned in connexion with the Smith process was a very high one for fuel oil to-day. A large amount of research work would have to be carried out on these oils before they could be refined profitably, and that work should be done before the processes were brought on the market.

Mr. E. B. EDWARDS, replying on behalf of the author, said that one of the differences between the Smith retort and the Del Monte retort was that the latter would not handle a swelling coal, whereas the Smith retort would handle coking or non-coking coals. The use of the carborundum linings was found necessary, owing to the high temperature in the heating flues; metal linings for the retorts would not withstand the temperature. By this process it was possible, by stirring the coal in the retort, to have a very high temperature in the flue and at the same time to have a low temperature in the retort itself. This gave a high capacity to the retort, which worked at the rate of 1 ton per hour continuously day in and day out. Another reason for the use of carborundum was its high conductivity. In reply to a statement by Dr. Ormandy, Mr. Edwards said that the fuel was now being used and sold in large quantities for the same purposes for which anthracite was used and had also been used in open grates and anthracite stoves in England. It was only after long scientific investigations and research work at the plant at Irvington, and based on its results, that the commercial plant at Clinchfield was erected. The trouble with low-temperature processes in England at the present time was that the processes were non-continuous, with the result that it was necessary to have low temperature in the flues, and it took about twelve hours to carbonise a charge of coal. The material must be agitated in order to release the gases and reduce the time of carbonisation; in America they were using a continuous retort in which the material was agitated by means of the paddles on the shaft. With regard to the question of the oils and their disposal, it was found in America that after distilling the tars, there was no difficulty in disposing of the oils. They had been able to get toluol and benzol by a very simple cracking process; in fact, the United States Government looked into the process during the war and gave a

grant for the purpose of installing a cracking plant. It was proved that toluol and benzol could be manufactured to meet with the Government requirements for making TNT and picric acid.

## Sydney, N.S.W., Section.

Meeting held at Sydney on November 10, 1920.

MR. E. W. CHALLINOR IN THE CHAIR.

### THE INFLUENCE OF SODIUM CHLORIDE AND SODIUM SULPHATE ON THE SOLUBILITY IN WATER OF SODIUM $\beta$ -NAPHTHALENESULPHONATE.

BY W. TERNENT COOKE.

The two systems water—sodium  $\beta$ -naphthalene-sulphonate—sodium chloride or sodium sulphate have been investigated over the temperature range 25°–65° C. at atmospheric pressure.

The sulphonate was made by heating together 1 g.-mol. of powdered naphthalene with slightly more than 1 g.-mol. of sulphuric acid (93%  $\text{H}_2\text{SO}_4$ ) to 180° C. for 3–4 hours. After cooling, and diluting to about 1 litre, 2 g.-mols. of common salt was added dissolved in about 1 litre of water. The precipitated sulphonate was fractionally crystallised until free from sulphate and chloride. It did not melt below 275° C. and was quite neutral to litmus. The sodium sulphate was used in the anhydrous form; the sodium chloride was Kahlbaum's purest fused salt.

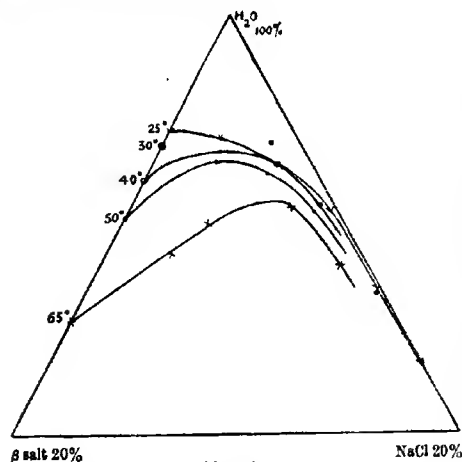


FIG. 1.

The two thermometers used were found not to differ by more than 0.1° from standards in the laboratory. Up to 50° C. the temperature was kept within 0.1°, and at 65° to within 0.2°.

Weighed quantities (0.2–5 g.) of sulphonate and of either sulphate or chloride were mixed with 10 c.c. of water in small glass vessels which were immersed in a thermostat and agitated for about 24 hours. Samples of the liquid were then withdrawn through a small filtering plug of cotton wool into a pipette jacketed with hot water, and the weights of the solution and of the salts left on evaporation were ascertained. The salts were dried at a temperature up to 150° C., trials having shown that the sulphonate is unaffected at this temperature. The dried salts were quantitatively analysed for sulphate and chloride, respectively, by precipi-

tation as barium sulphate, or by titration with standardised silver nitrate solution, using chromate as indicator. Trials with known amounts showed that both these methods are accurate when sodium chloride, or sulphate, is mixed with an equal weight of sulphonate. Incidentally, the solubility of the sulphonate alone had to be determined as only one value, that at 23.9° C., could be found (Thorpe, Dict. Appl. Chem., 1912, III, 574). The solubilities of the sulphate and chloride were taken from Landolt and Börnstein's tables.

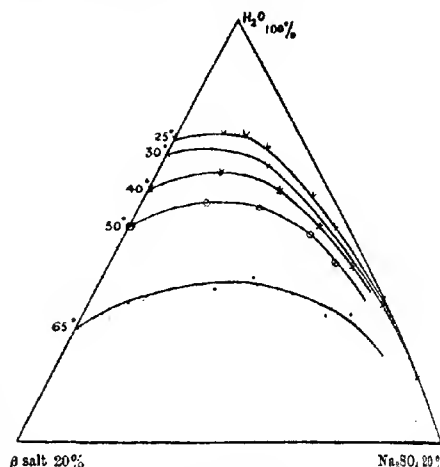


FIG. 2.

In one experiment a mixture of 1.5 g. of  $\beta$  salt, 0.3 g. of sodium chloride, and 10 c.c. of water was shaken together for 24 hours at 65° C.; 5.2338 g. of solution was filtered off, and this weight of liquid left 0.6003 g. of mixed salts on evaporation. These salts required silver nitrate solution equivalent to 0.1544 g. of NaCl. Therefore 0.4459 g. of  $\beta$  salt was present. The solution contained therefore:—Water 4.6665 g. (88.57%),  $\beta$  salt 0.4459 g. (8.47%), sodium chloride 0.1544 g. (2.93%). The results obtained are shown in the accompanying diagrams.

The curves show that the solubility of the  $\beta$  salt is reduced practically to zero when the concentration of the inorganic salt reaches about 10 g. in 100 g. of solution, especially at lower temperatures. The figures are derived from experiments made by using the  $\beta$  salt in excess and gradually increasing the amount of inorganic salt. Applying the ordinary method of graphical analysis to this three-component system, and from the appearance of the insoluble residues, the residue in each case is found to be the organic salt. The curves, therefore, represent the solubilities of the organic salt in solutions of the inorganic salts. An attempt was made, at 25°, to find the point of intersection of the curve shown in the figure with the curve representing the solubility of sodium sulphate in presence of the  $\beta$  salt. Two experiments were made at 25° using excess of both salts. The evaporated filtrates left 1.1444 and 1.302 g. of residue respectively, which residues yielded barium sulphate equivalent to 1.1284 and 1.2335 g. of  $\text{Na}_2\text{SO}_4$  respectively, showing that the influence of the  $\beta$  salt on the solubility of the sulphate is practically negligible. The point of intersection lies, therefore, practically on the solubility curve of the sulphate.

The above work was carried out in the Chemical Department of the University of Queensland, and I desire to thank the authorities of that University, and especially Prof. B. D. Steele, for granting me every facility for work. I am also indebted to Prof. Steele for the interest he took in the research.

The University, Adelaide.

## Communications.

### THE ESTIMATION OF COCONUT AND PALM-KERNEL OILS IN FAT MIXTURES IN WHICH BOTH OILS MAY BE PRESENT.

BY W. N. STOKOE, B.SC., A.I.C.

It is common knowledge that the estimation of coconut and palm-kernel oils in mixtures when both are present is a matter of great difficulty owing to the similarity in composition of the two oils. Coconut oil, however, is richer than palm-kernel oil in the lower fatty acids, as is indicated by its higher Polenske value. Further, the insoluble fatty acids from coconut oil which distil over in the Polenske test are liquid at room temperature, whereas the volatile acids obtained from palm-kernel oil under the same conditions are solid. This difference suggested a possible method for the identification and estimation of the respective oils.

Several methods for the estimation of coconut and palm-kernel oils in mixtures have been described in the literature. Burnett and Revis (Analyst, 1913, 38, 255) have devised a method which depends on the relative solubility of the barium salts of the insoluble volatile acids in alcohol of definite strength. The temperature of turbidity on cooling is observed, palm-kernel oil giving a higher figure than coconut oil. Mixtures of the two oils are stated to give "turbidity temperatures" proportionately intermediate between the extreme values given by the pure oils. This method is expensive owing to the quantity of strong alcohol required, and has the disadvantage that the solution of the barium salts in alcohol is seldom perfectly clear, and the exact "turbidity point" is often difficult to judge accurately. A second method devised by Blichfeldt (J., 1919, 151 r) depending on the m.p. of the insoluble volatile acids has been published; in the preliminary work preceding this investigation a modification of this method was tried, but without success. After further experiment the following method was devised.

In the standard Reichert-Polenske process, after distilling 110 c.c. the liquid is cooled and the insoluble acids collected on a filter; the condenser tube is rinsed down with 20 c.c. of warm water (30°–40° C.), which is poured over the filter. The filter paper is washed with warm water (30°–40° C.) and as soon as the last drop of water has drained through several capillary tubes (internal diameter of 1 mm.) are filled to the depth of  $\frac{1}{2}$  in. with the now melted acids. Two of the tubes are immediately attached, one on either side, by means of a small rubber band, to a thermometer graduated to read  $1/10^\circ$ , and the thermometer fitted into a test tube by means of a cork having a hole through which the thermometer is placed. The tube is supported in a beaker containing ether, the surface of which should be above the level of the acids in the capillary tubes. A rough idea as to whether the bulk of the acids is from palm-kernel or coconut will have been obtained from the appearance of the acids in the original Reichert-Polenske distillate. If the acids are solid at ordinary temperature it is necessary to warm the ether to about 30° C. A gentle stream of air is forced through the ether by means of a foot bellows so as to lower its temperature gradually, the rate of cooling being so adjusted that the temperature of the ether is 2° C. below that registered by the capillary tube thermometer. As the cooling progresses the fatty acids will become slightly cloudy, then a distinct "seeding" or crystallising will occur. At the first appearance of crystals the temperature is noted. This "seeding point" is quite definite and sharp. At this point the acids in the capillary tubes appear liquid with a number of tiny white crystals along

the sides of the tube. It is important in performing the test that the rate of cooling should be regular.

The following tables show results obtained by this method. As was to be expected, there is a slight variation between the figures obtained for samples of different origin. The oils used in the mixtures were made by mixing equal quantities of the different individual samples in order to obtain an average result:—

#### Mixtures containing only coconut and palm-kernel oils.

Coconut.	Palm-kernel.	"Seeding" point, °C.	Average.
%	%		
85	15	{ 11.8 11.6 11.6 11.9 11.6 12.0 13.5 13.4 13.2 }	11.8°
75	25	{ 13.2 13.5 13.2 13.2 13.5 13.2 15.7 15.3 15.6 }	13.4°
60	40	{ 15.4 15.3 15.3 15.4 15.3 15.3 16.9 17.0 }	15.5°
50	50	{ 16.8 17.0 17.9 17.9 17.9 18.0 }	16.0°
40	60	{ 19.6 19.4 19.4 19.5 21.6 22.0 }	17.9°
25	75	{ 21.7 22.0 21.7 22.0 21.7 22.0 }	19.5°
10	90	{ 21.7 22.0 21.7 22.0 21.7 22.0 }	21.8°

Four samples of coconut oil of different origin and the same number of palm-kernel oil were examined. The highest figure for the "seeding point" of coconut oil acids was 11.4° C. and the lowest 9.9°; average 10.75°. The figures for palm-kernel acids were: highest, 23.2°; lowest, 22.05°; average, 22.75° C.

#### Mixed fats containing 80% oleo, 10% cottonseed oil, 60% coconut or palm-kernel oil or mixtures of them.

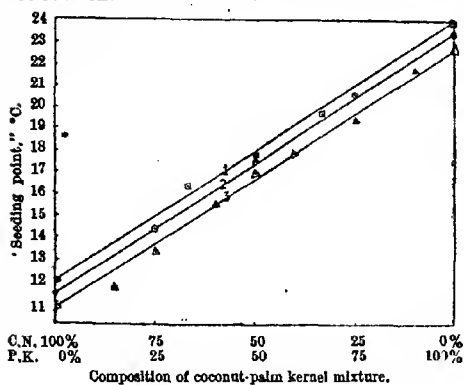
Relative proportions of coconut and palm-kernel oils	"Seeding" point, °C.	Mean.
Coconut 100% .. ..	{ 11.3 11.6 11.3 11.3 11.5 11.4 23.5 23.7 23.5 23.7 }	11.4°
Palm-kernel 100% .. ..	{ 14.5 14.0 14.5 14.1 17.6 17.6 17.5 17.5 }	23.6°
Coconut 75%, palm-kernel 25%	{ 14.5 14.1 14.5 14.1 17.6 17.6 17.5 17.5 }	14.3°
Coconut 50%, palm-kernel 50%	{ 20.4 20.9 20.4 21.0 20.4 20.3 }	17.55°
Coconut 25%, palm-kernel 75%	{ 20.4 20.9 20.4 21.0 20.4 20.3 }	20.6°

#### Mixed fats containing 50% oleo, 20% cottonseed oil, 30% coconut or palm-kernel oil or mixtures of them.

Relative proportions of coconut and palm-kernel oils	"Seeding" point, °C.	Mean.
Coconut 100% .. ..	{ 12.0 12.0 12.0 11.9 12.0 12.0 24.0 24.0 24.0 24.1 }	12.0°
Palm-kernel 100% .. ..	{ 20.0 19.6 19.9 20.1 19.5 19.8 17.8 17.2 17.8 18.2 17.3 17.8 }	24.0°
Coconut 33½%, palm-kernel 66½%	{ 16.6 16.2 16.2 16.4 16.2 16.4 }	19.8°
Coconut 50%, palm-kernel 50%	{ 16.6 16.2 16.2 16.4 16.2 16.4 }	17.7°
Coconut 66½%, palm-kernel 33½%	{ 16.6 16.2 16.2 16.4 16.2 16.4 }	16.3°

It will be observed that the presence of other fats raises the "seeding point," but that, roughly speaking, parallel lines are given on the graph. Thus in the analysis of an unknown fat mixture it will be necessary first to ascertain the approximate quantity of the coconut group of fats present by determining the saponification value and the Reichert-Polenske value. Reference is then made

to the corresponding line or intermediate line if necessary on the graph diagram, and from this the relative proportions of coconut and palm-kernel oils are read off.



The method can be readily carried out as an extension of the standard Reichert-Polenske method, the contents of the capillary tubes being added to the alcoholic solution of the remainder of the insoluble acids and the whole being titrated as usual to obtain the Polenske value.

The following table shows results obtained on margarine samples of known composition:—

Relative proportion of C.N. & P.K. in sample.	Sapon. value.	Reichert-Meisels.	Polenske.	"Seeding point" °C.	Relative proportion of C.N. & P.K. indicated.
Palm-kernel 100%	228.7	4.05	6.1	23.5	100% P.K.
Palm-kernel 100%	208.2	1.36	1.3	24.0	100% P.K.
Coconut 100%	223.7	5.01	6.40	11.8	100% C.N.
Coconut 100%	246.3	7.15	13.0	11.6	100% C.N.
Coconut 50% palm-kernel 50%	244.1	5.61	11.2	16.9	51% C.N., 49% P.K.
Coconut 25% palm-kernel 75%	229.1	4.35	7.30	20.6	28% C.N., 72% P.K.

Laboratory, Craigmillar Creamery Co., Ltd.,  
Craigmillar.

## HYDROGENATION IN THE NAPHTHALENE SERIES.

BY F. M. ROWE.

A communication on dihydronaphthalene by Straus and Lemmel (Ber., 1921, 54, 25) has drawn my attention to an error inadvertently introduced into my paper on this subject (J., 1920, 241 r) when it was written from the laboratory note-books. It is stated that both 1.4- and 1.2-dihydronaphthalenes were purified by means of their mercuric acetate compounds, whereas actually it was only the former of these compounds that was purified in this manner. My assistant, Miss Levin, attempted to purify 1.2-dihydronaphthalene by this means, but found that the mercury compound formed, of high melting point and insoluble in boiling benzene, did not yield the hydrocarbon when treated with acid, although this compound was not examined further at the time. Straus and Lemmel have now found that mercuric acetate exerts an oxidising action on 1.2-dihydronaphthalene, forming 1.2-dihydroxy-tetrahydronaphthalene, and that the insoluble mercury compound is  $\text{Hg.O.CO.OH}$ .

The only satisfactory method by which the purity of 1.2-dihydronaphthalene may be ensured consists in its conversion into the dibromide, which is purified by recrystallisation, followed by the removal of the bromine atoms.

## THE VULCANISATION OF RUBBER.

In a letter dated January 22 to the Editor (see also India-Rubber J., 1921, 61, 177), M. A. Dubosc claims that he had already anticipated Peachey's discovery of the possibility of vulcanising rubber by the action of sulphur dioxide and hydrogen sulphide at the ordinary temperature (see Peachey and Skipsey, J., 1921, 5 r). In 1915 (cf. India-Rubber J., May 1, 1915) M. Dubosc had suggested that, of the various allotropic modifications of sulphur, only the colloidal form is able to vulcanise rubber. Chemically pure rubber is known to undergo vulcanisation only with great difficulty, the presence of the natural resinous and protein impurities being essential to the change, which is also aided by the addition of metallic oxides such as magnesium oxide or litharge. M. Dubosc's view of the vulcanisation process was that the resinous matter first reacts with part of the ordinary sulphur present, with formation of hydrogen sulphide, whilst any oxidised rubber or metallic oxide in a similar manner gives rise to sulphur dioxide; these gases, in solution in the rubber, then react with formation of water and sulphur, the latter being produced in the active "monatomic or colloidal" condition capable of effecting vulcanisation.

Mr. Peachey (India-Rubber J., 1921, 61, 163) replies that the new process is one of "cold vulcanisation" operating at the ordinary temperature, whereas the above theory referred to the ordinary process of hot vulcanisation; in a critical review of M. Dubosc's claim, he questions the accuracy of certain of the assumptions and directs attention to the facts that not only would any sulphur present during hot vulcanisation be in the molten condition, but that for the production of a pure ebonite containing 32% of combined sulphur, the volume of sulphur dioxide and hydrogen sulphide necessary according to above theory would be approximately 100 and 200 times respectively that of the rubber.

## THE EROSION OF BRONZE PROPELLERS.

(See J., 1921, 38—45 r.)

Colonel N. BELAIEV wrote that he had been particularly struck by the similarity between the erosion of propellers and the erosion of guns. In both cases the process of erosion seemed to be divided into two phases—the preliminary, and the final. In the case of a propeller, this preliminary phase, according to Dr. Silherrat, consisted in the "water-hammer" action of water broken by vacuum bubbles; in the case of a gun, according to Tschernoff (Proc. Int. Assoc. Testing Materials, 1912, 2, A, 121), it consisted in the thermal action of the gases on a thin film of the metal on the internal surface of the gun-tube. The next stage, or the actual erosion, in both cases was due to mechanical causes only, being caused either by frictional rub of the water (on the propeller), or by the frictional rub of the particles of the gases (as in guns) forcing their way out of the tube. He (Colonel Belaiev) felt inclined, therefore, to endorse the author's view that after the surface of the propeller had been damaged by any of these agencies, further erosion was chiefly caused by the frictional rub of the water. He also thought that, just as in guns, the influence of structure was considerable, and that whilst, according to the Committee report, there might be no "connexion between the structure of a metal and the distribution of erosion," the wearing away of the metal was largely determined by the structure of the metal. The problem of structure was more complex in guns, where the gases had a certain hardening effect, and where the initial structure might therefore be altered considerably; in propellers the position was more simple, and only resolved itself into the question, as the author rightly pointed out, of finding an alloy with the most suitable initial structure.

## Sydney Section.

## THE PHOSPHATES OF NAURU AND OCEAN ISLANDS.

BY THOMAS STEEL.

The only detailed analyses of different types of the phosphates from these islands with which the author is acquainted have been published by C. Anderson (Records Australian Museum, Sydney, 1903, 5, 13), who gives descriptions of ten specimens, mostly from Ocean Island, with analyses of four. Anderson's descriptions and analyses agree well with some of those detailed in this paper. F. R. C. Reed (Geol. Mag., 1903, 10, 298) describes the physical appearance of a series of specimens from Ocean Island, which agree precisely with several of the author's samples, and gives an analysis by Voelcker of a specimen described as "hard rock," which shows 4.5% of magnesia. As will be seen from the descriptions following, there is a considerable variety of types. For the specimens, which were selected with the special view of being representative, the author is indebted to his brother, Mr. F. W. Steel, who was resident on Nauru for some time. Most of the samples are from Nauru, but the Ocean Island phosphates are of precisely similar character.

## Analysis of the phosphates.

	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16.
Water at 200° C. ..	1.75	1.02	1.70	1.28	0.93	1.18	2.02	3.50	4.88	4.23	0.70	1.83	1.60	0.27	0.30	1.05
Organic matter* ..	1.93	1.85	1.88	2.73	1.46	1.78	3.53	1.47	2.35	1.62	0.38	2.00	2.70	0.58	0.70	1.33
Sand ..	0.07	0.10	0.15	0.10	—	—	0.05	0.05	0.03	—	0.30	0.23	0.20	0.10	0.04	0.15
Lime (CaO) ..	55.20	55.10	54.80	53.10	55.80	55.20	54.80	55.80	52.60	53.20	56.40	54.40	54.70	33.90	34.60	41.70
Magnesia (MgO) ..	0.43	0.36	0.50	1.30	0.29	0.16	0.29	0.29	0.43	0.47	0.23	0.11	Trace	26.61	19.93	13.66
Sulphuric anhydride (SO <sub>3</sub> ) ..	0.27	0.37	0.45	0.33	0.47	0.17	0.14	0.07	0.14	0.14	0.17	0.17	0.07	0.07	0.21	0.58
Phosphoric oxide (P <sub>2</sub> O <sub>5</sub> ) ..	38.64	37.57	37.57	31.76	39.30	37.04	38.52	39.30	39.07	37.94	40.44	38.77	39.03	0.16	0.25	5.70
Carbon dioxide (CO <sub>2</sub> ) ..	1.99	2.77	2.77	8.05	1.12	1.67	0.67	1.00	0.95	2.48	0.65	1.80	1.36	43.30	44.75	37.10
	100.33	99.74	99.82	99.20	99.87	98.10	100.97	101.48	100.65	100.08	99.26	99.31	98.63	100.96	100.98	101.27
*Nitrogen ..	0.05	0.04	0.05	0.03	0.01	0.01	0.11	—	0.01	0.08	0.01	0.02	0.05	0.02	0.01	0.01
Ammonia by distillation with lime ..			0.0085				0.0085									

## Description of specimens.

1. A beautiful, compact, pale grey rock of uniform very fine texture, closely resembling the porcelain of which Wedgewood mortars are made. Fracture conchoidal, not stratified.

2. Extremely porous, sintered mass in which coral structure is well preserved, the vertical ribs in the coral tubes being clearly defined. Bears a strong resemblance to a mass of calcified sponge.

3. Somewhat porous, in shape like coarse branching coral, but no organic structure could be detected. Some specimens are hollow, the interior being thickly lined with interlacing vermiform processes, whilst others are solid, being filled with a mixture of similar processes and of oolitic grains.

4. A breccia, consisting of small oolitic grains and rough angular fragments loosely cemented.

5. Similar to 1, but in fine layers of dark and light grey.

6. Coarse and fine loose water-worn grains and pebbles. May be described as a mixture of pisolitic and oolitic grains; grey.

7. Similar to 6, but the grains are black, being coated with a skin of humus easily rubbed off. This constitutes the surface "soil," 4 to 6 inches in depth. Ocean Island.

8. Compact, surface smooth, somewhat resembling grey flint.

9. Thin, brown layers like banded agate, dark and light brown. Very hard and dense. Breaks with a splintery fracture.

10. This sample bears a striking resemblance to a mass of commercial gum benzoin. Brown, with angular white enclosures.

11. Black, compact trachyte-like, of oolitic structure. Bears a strong superficial resemblance to a piece of dolerite. Weathers white on outer surface.

12. A hard, grey, massive rock, having numerous vesicles lined with brown material, like 10, and small spheres of same substance.

13. Resembles coarse, grey sandstone, composed of oolitic grains loosely cemented.

14. Like dense limestone, with numerous large white shell casts.

15. Fine columnar structure of coral, adherent to dense limestone. Columns about  $\frac{1}{4}$  inch in diameter, with vertical radial septa dividing the tube into a series of sectors. Coral part only taken for analysis.

16. Buff and white diffusely banded, compact rock, dense and hard. From reef. Ocean Island.

17. Small sample, showing in vertical section flattened double coral tubes, each element of the double tube being about  $\frac{1}{16}$  inch in diameter. The

septa of the tubes are very prominent and well preserved, forming a series of superimposed closed cells, in depth about equal to their diameter. Not analysed, but high in phosphoric acid.

Unless otherwise stated, all the samples are from Nauru Island.

Only traces of iron or alumina were present in any of the samples. Varying small amounts of fluorine occur in many of the samples.

The original source of the phosphoric acid is generally conceded to have been the accumulations of guano from the colonies of sea-birds using the islands as nesting places.

Most of the samples bear evidence of having undergone considerable metamorphosis and of having been deposited either from solution or as sediment. Only two samples, 2 and 17, of those high in phosphoric acid, retain any organic structure. Of the others, 14 and 15 exhibit the remains of shells and coral respectively, but these are dolomites containing only a small proportion of phosphoric acid.

The four samples analysed by Anderson are high in phosphoric acid, and of these two are described as displaying organic structure, coral, or shell.

It is not possible to offer any opinion as to the extent to which coral has entered in the course of formation of the various types described above, but it is possible that the deposits devoid of organic structure may have been formed direct from guano without the intervention of coral. When dead coral

is submerged in sea water it becomes dolomitised by reaction between the calcium carbonate of the coral and magnesium chloride of the water, producing such samples as 14 and 15. Where the supporting rock consisted of such dolomite, the phosphatic basis of the guano, after the loss of the organic constituents by decay and leaching, may have undergone transformation by solution and redistribution without in any way affecting the inert underlying rock. Doubtless carbon dioxide in percolating rain water would materially assist in the transformation of the phosphate.

Fresh coral has been reported to contain from 0.28 to 0.84% of calcium phosphate. Fresh coral is stated (Watts' Dict. Chem., II., p. 84) to contain about 1% of magnesia and 2.1–9.4% of organic matter, the remainder being calcium carbonate.

The analysis of the above phosphates show that part only of the phosphoric acid is present as calcium phosphate ( $\text{Ca}_3\text{P}_2\text{O}_8$ ), the relative proportions of constituents indicating the presence of a varying proportion of tetracalcium phosphate ( $\text{Ca}_4\text{P}_2\text{O}_{10}$ ). The author has observed this in other natural phosphates, and, quite recently, in the deposit of "tartar" from the teeth of man, sheep, ox, and other animals (Steel, Proc. Linnæan Soc. N.S. Wales, 1920, 324).

The question of origin was the subject of an important lawsuit in Melbourne some years ago. A firm of chemical manufacturers claimed that a cargo of phosphate from Ocean Island should be classed as guano owing to its reputed bird origin. Guano was admitted free of wharfage, whereas a charge of one shilling per ton was levied by the Harbour Trust on minerals, under which category they placed the "rock phosphate" of Ocean Island. The verdict of the court was in favour of the Harbour Trust.

Mr. F. W. Steel has furnished the following notes from his observations when resident on Nauru:—

"The substructure of both islands is hard dolomite without trace of coralline structure, containing embedded fossil shells. The coral pinnacles among which the phosphate deposit is found come up to the present surface and in a number of places project beyond to a considerable height, notably two large masses in the centre of Nauru, which attain a height of about 100 ft. above ground level. The highest point of the island of Nauru is stated to be about 250 ft. above sea-level. On Nauru are a number of caves in the dolomite rock; these are evidently shrinkage cracks and show but slight stalagmitic structure. In places the surface layer of the dolomite seems to have been partly phosphatised, but this may be merely a layer of phosphate deposited thereon from solution.

Around Ocean Island is a raised beach or rough flat of very limited extent, where in places are numerous pinnacle masses of coral, evidently the remains of the coral structure, and which are very rough and sharp through weathering. The pinnacles embedded in the phosphate, on the other hand, show smooth surfaces with layers of phosphatised coral. It is extremely probable that the shore pinnacles were originally surrounded by phosphate, which has been subsequently washed out by the sea, as on the flat reef on Ocean Island there are numerous 'pipes' of phosphate of pisolitic structure projecting from the surface, the grains being cemented together with phosphate. These, being formed by the filling in of 'blowholes' in the coral and being harder than the latter, have remained when the surrounding coral was removed by wave action. The 'pipes' stand up to as much as three feet above the reef level. The flat reef is submerged at each high tide. The sand of the beaches is all coral debris, shells, etc."

## Yorkshire Section.

Meeting held at Queen's Hotel, Leeds, on  
January 17, 1921.

DR. L. L. LLOYD IN THE CHAIR.

### THE PRESENCE OF NITRATES IN THE LEAVES OF TREES GROWING NEAR PICRIC ACID WORKS.

BY B. A. BURRELL, F.I.C., AND G. W. DOUGLAS.

In the production of picric acid by the nitration of phenolsulphonic acid large quantities of oxides of nitrogen are evolved. During the war considerable volumes of these gases sometimes escaped from the nitrating sheds to the detriment of any vegetation that might be in the vicinity.

In 1916 one of us was consulted in respect to such damage, and during that and subsequent years we examined numerous samples of leaves taken from trees growing near to a large picric acid works.

When the wind was blowing from the works to the agricultural land (about  $\frac{1}{2}$  a mile distant) on which the damage had and was taking place, the distinctive smell of the nitric fumes was easily perceptible.

The district was well wooded and the damaged trees could be easily recognised, the leaves being dry, shrivelled, and falling readily. When the trees were in clumps, those nearest to the works showed the most damage, those behind being to a certain extent protected. Where the trees were growing behind a wall situated between them and the works, the portion immediately behind the wall did not show material damage, whereas that portion which projected above the wall and to which the gases could obtain direct access was obviously damaged.

The beeches appeared to suffer the most, sycamores, elms, and oaks being more resistant. A certain amount of sulphuric acid vapour also escaped from the works from time to time, but not to anything like the same extent as the oxides of nitrogen. The sulphurous vapour would no doubt contribute to the damage, but this is more difficult to determine as sulphates occur naturally in all vegetation\* whereas nitrates do not though according to Wiley they occur in considerable quantity in the tobacco plant.† In this connexion it has been stated that the tobacco plant is particularly injured by nitric acid, and that next to all herbaceous plants, trees generally suffer.‡

Our experiments were made on leaves which had been air dried in a room where they could not come into contact with nitric fumes.

Ten grams of the leaves, free from twigs &c., were placed in a clean dry stoppered bottle, 200 c.c. of distilled water was added, and the bottle and contents were gently agitated at frequent intervals for  $\frac{1}{2}$  hr., when the aqueous extract was poured off, the leaves remaining in the bottle were drained for a specified time, the drainings being added to the decanted liquid and the bulk made up to 200 c.c. Known quantities (generally 10 c.c.) were taken for the detection and estimation of nitrates, only those methods being used which were capable of estimating minute quantities of nitrates in the presence of comparatively large quantities of nitrogenous organic matter.

\* R. R. Tatlock and R. T. Thomson, Analyst, 1914, 39, 203-210.  
† E. Ebermayer, J., 1899, 282.  
‡ Harvey W. Wiley, Principles and Practice of Agricultural Analysis, Vol. III., pp. 598, 599, 1897.  
§ A. W. and M. W. Blyth, "Poisons," 1906 4th Ed., p. 110.



**Phenolsulphonic acid method.**—There was no difficulty in detecting the presence of nitrates, but for quantitative purposes the process was not satisfactory, the colour produced being of a much browner tint than that of the standard ammonium nitrate solution, and when the solutions were allowed to stand for a short time a greenish tint began to develop which made comparison still more difficult.

**Indigo method.**—This was also found unreliable for quantitative purposes.

**Brucine method.**—The results quoted were all obtained by this method, which was found to be well suited for the purpose. The process is due to West Knights,<sup>1</sup> and is based upon the well-known reaction of brucine with nitrates, a deep red colour being produced on evaporation to dryness in the presence of a small quantity of oxalic acid. The residue thus obtained is dissolved in a small quantity of water, the solution filtered, and the colour compared with that of a solution containing a known quantity of nitrate.

A standard solution of potassium nitrate is prepared by dissolving 0.721 g. in distilled water and making up to a litre: each c.c. of this solution is thus equal to 0.0001 g. of nitric nitrogen. 10 c.c. of the solution is evaporated to dryness on a water bath and 3 c.c. of a 1% solution of brucine in alcohol and 0.5 c.c. of a saturated solution of oxalic acid are added to the residue, which is again evaporated to dryness. A little distilled water is then added to dissolve the residue and the volume made up to 100 c.c. Each c.c. of this solution = 0.00001 g. of nitric nitrogen.

One example of the damaged leaves of a sycamore may be given. The extract from 0.5 g. of leaves was treated as described, the final solution being filtered to remove any calcium oxalate and then diluted to 50 c.c. in a Nessler cylinder; 7 c.c. of the standard red solution was required to match the colour given by 10 c.c. of leaf extract. Then  $0.00001 \times 7 \times 200 = 0.014\%$  of nitric nitrogen.

The following is a list of results obtained:—

TABLE A.

No.	Tree	Location	Nitric N. %	Nitric acid %
No. 1	Beech (damaged)	...	0.056	0.232
" 2	Elm	1 mile from works	0.072	0.324
" 3	Elm	near to No. 2	0.012	0.054
" 3a	Sycamore	Leaves fallen to the ground	0.060	0.270
" 4	Beech	...	0.050	0.225
" 5	Sycamore	...	0.084	0.378
" 6	Ash	...	0.021	0.108
" 6a	Grass	From edge of man-hole from which nitric fumes escaped at intervals	0.032	0.144
" 7	Beech	...	0.036	0.162
" A	Beech (damaged side)	...	0.018	0.081
" A1	Beech (same tree as A, undamaged side)	...	—	—

A sycamore, an elm, and an ash from the same estate, but some distance from the works and which from their position could not have been exposed to the nitric fumes, were all undamaged and did not contain any traces of nitrates. (These samples were taken in August, 1916.)

TABLE B.

Results in parts per 100. (Samples taken July, 1918.)  
These trees are not included in Table A.

No.	Tree	Nitric N. %	Total Chlorine %	Sulphur acid. %	Trioxide %
No. 1	Sycamore (damaged)	0.014	0.799	0.62	0.78
" 2	" (undamaged)	—	0.517	0.38	0.46
" 3	Elm (damaged)	0.044	1.17	0.96	1.12
" 4	Elm (undamaged)	—	0.90	0.63	0.38

\* Total acid expressed in terms of sulphuric acid.

The damaged and undamaged sycamores were 12–15 yards apart, while Nos. 3 and 4 were from

<sup>1</sup> Analyst, 1881, 56-58.

the same tree, No. 3 being exposed to fumes, No. 4 being protected by a wall.

In addition we have tested the leaves from elm, elder, hawthorn, and plane trees growing in Springfield Mount, Leeds, and from ash, beech, elm, and sycamore trees growing at Ilkley, and have in all cases proved the absence of nitrates. In none of the leaves, either damaged or undamaged, could we find any trace of nitrates.

#### DISCUSSION.

Mr. W. McD. MACKAY suggested that the effect of fumes on a leaf would depend on the surface of the latter; he would expect a smooth leaf to be more immune than one with a rough surface. He asked if the shrivelling effect observed might not have been due as much to sulphur oxides as to the oxides of nitrogen.

Mr. BURRILL, in reply, said that the difficulty with regard to sulphuric anhydride was that all leaves contained an appreciable quantity of sulphate, and no definite line could be drawn between the natural amount and an abnormal amount sufficient to cause damage. No such difficulty occurred with nitrates.

## Communications.

### THE ACTION OF HYDRAZINES ON CELLULOSE ACETATES.

BY W. LEIGH BARNETT, B.Sc., A.I.C.

The object of this investigation was to determine whether phenylhydrazine and *p*-bromophenylhydrazine form any definite chemical compounds with cellulose esters, which compounds might be comparable with simpler hydrazones. Under normal conditions cellulose has not been known to react with phenylhydrazine to give well-defined products, owing no doubt to the inertness of the cellulose complex, and to the fact that it is insoluble in ordinary solvents which are used in the case of simpler ketones. Again, it is possible that the cellulose complex is held together by oxygen linkages, and until these linkages are broken and the cellulose complex degraded the ketonic or aldehydic groups cannot function as such. Consequently no phenylhydrazones of cellulose are formed by reason of the enforced inactivity of these latent groups. It was considered that the problem could be attacked by first of all forming cellulose esters, made by the author's method (J., 1921, 87), so as to ensure the minimum of degradation of the complex, and then treating these esters with the hydrazine. The fact that these esters are readily soluble in many solvents obviously simplifies the problem. It is clearly of importance that the cellulose esters utilised in this research should be made in such a way as to preclude the possibility of dissociation of the parent complex, otherwise the initial ester, being of lesser magnitude than the cellulose complex, would show an enhanced reactivity due to the degradation which it had undergone.

If dissociation of the cellulose complex,  $(C_6H_7O_2)_n$ , occurs, and if this is brought about by rupture at the oxygen linkages freeing more and more ketonic groups as the value  $n$  grows less, then at the same time there will be a progressive increase in the amount of phenylhydrazine reacting with the ketonic group to form hydrazone. Taking the case of cellulose triacetate, which can be written as  $[C_6H_7O_2(CH_3CO_2)_3]_n$ , and supposing for any value of  $n$  there be one ketone group only, the complex can be written in the form,  $C_{6n}H_{7n}O_{m-1}O$   $(CH_3CO_2)_n$ , and the corresponding phenylhydra-

azone will be  $C_{12}H_{17}O_{m-1}C_6H_5NH.N(CH_3CO)_n$ . In the case of the *p*-bromophenylhydrazones the general formula will be similar as shown by  $C_{12}H_{15}BrO_{m-1}C_6H_4BrN_2H(CH_3CO)_n$ . The nitrogen and also the bromine content of these compounds will vary inversely as the value of *n*. Conversely, if by experiment the least amount of bromine, and nitrogen, be ascertained, and if these quantities be definite together with the properties of the specific compound under observation, irrespective of the mode of preparation, it should be possible to fix on a value for *n*, and hence get some indication of the molecular complexity of the cellulose complex.

The results obtained from a large number of experiments both with di- and triacetylcellulose, and also phenylhydrazine and *p*-bromophenylhydrazine, indicate that it is possible to obtain a series of definite compounds containing nitrogen (and halogen) in quantities corresponding with the amounts expected from cellulose acetates calculated according to the general formulae given above, provided that the value *n* be chosen suitably. These hydrazones have definite melting points, and also exhibit the normal characteristics of ketose hydrazones. The experimental methods adopted were such as to eliminate the possibility of the purified products being merely adsorption products of the hydrazine by the cellulose ester colloid. Essentially the method consisted in treating a solution of the cellulose acetate in a suitable solvent with the hydrazine, under different conditions of temperature and time of duration of the treatment, followed by removal of the solvent and excess hydrazine, and then exhaustive purification of the product by repeated solution, precipitation, filtration, and washing, the number of re-precipitations of the substance depending on its properties.

The following solvents were employed for the initial solution of the cellulose acetate:—Aniline, chloroform, glacial acetic acid, *o*-toluidine, nitrobenzene, pyridine, and phenol. The cellulose acetates used were made by the author's method (*loc. cit.*), from filter paper, cotton wool, and bleached cotton yarn.

As important differences exist between acetylcellulose phenylhydrazones and the bromophenylhydrazones they will be treated separately.

**Acetylcellulose phenylhydrazones.**—It was found possible to obtain the same acetylcellulose phenylhydrazones starting from different cellulose triacetates, irrespective of the initial properties, and also of the mode of operation in the preparation of the hydrazones. The final products form, as a rule, amorphous powders, which may be coloured from a whitish-grey to a very pale yellow or light brown. All these substances are soluble in hot alcohol, but in different degrees. The solubility is apparently influenced by the solvent employed in the first part of the reaction. The temperature at which the treatment of the cellulose acetate with the phenylhydrazine is carried out has less influence on the nature of the product than the solvent, and to a lesser extent the time of heating. An important physical difference between the phenylhydrazones obtained from the triacetate from paper and cotton was noticed. If the product in the form of an amorphous powder, which is not perfectly freed from traces of alcohol used in the final washing in the purification process, be placed in an oven to dry at 100° C., the whole mass rapidly coalesces and assumes the appearance of a transparent yellow resin. These resinous masses are hard, brittle, and flaky. In alcohol they give solutions similar to shellac varnishes. Evaporation of the alcohol from thin films of the solution leaves hard, glossy, transparent, but somewhat brittle, films. Whether the product be in the form of an amorphous powder or resin, it possesses a well-marked melting point, at which unstable viscous brown liquids are formed.

The melting points appear to be associated with the solubility in alcohol, and not with the complexity of the compound, as deduced from the nitrogen content. Thus the most soluble product obtained (1.02% N, m.p. 229° C.) was of the resinous type, and was made from a filter paper cellulose triacetate. The next in order of solubility had 4.16% N, m.p. 238° C. Several compounds less soluble in alcohol melted at 245° C., although the nitrogen content varied between 0.76 and 4.44%, the same samples giving concordant figures. The least soluble products melted about 262° C., the nitrogen content being 0.90–1.78%. All these compounds dissolve easily in acetone, giving clear solutions from which they can be precipitated unchanged by suitable means. This latter fact shows that there is no possibility of these substances being adsorption products, otherwise the acetone would react with the liberated phenylhydrazine in the usual manner.

In the case in which chloroform is used as a solvent for the cellulose acetate, followed by digestion with phenylhydrazine, the reaction is complicated by the formation of a crystalline substance which separates in very fine, glistening plates. This was found to be due to the action of the chloroform on the hydrazine. Steps had therefore to be taken to eliminate the possibility of contamination of the cellulose derivative by this crystalline substance, which was separated and investigated separately. (Details of this substance will be given in a separate paper.)

**Experimental.**—The method adopted consisted essentially in first dissolving the cellulose acetate in the chosen solvent, which was generally warmed to facilitate solution, and when complete solution had occurred the phenylhydrazine was added, after which the mixture was heated to the boiling point for known periods of time. The solvent was then removed by suitable means depending on its nature. In the case of chloroform this was effected by mixing with excess of water and distilling off the chloroform on a water-bath. In the case of aniline, nitrobenzene, etc., after mixing with water the excess of solvent was removed by steam distillation. The remaining products were filtered off, washed, pressed, and then repeatedly dissolved in hot alcohol (under a reflux condenser), followed by cooling to precipitate the substance in a fine powdery form, filtration, washing with alcohol, and then redissolving in more alcohol, and re-precipitation. This process was repeated until the melting point of the product was constant. Generally from eight to twelve re-precipitations from alcohol were employed, the number depending on the nature of the product, and its solubility in alcohol. In some cases it is necessary to use mixtures of alcohol and ether or of alcohol and water for the final stages in the purification.

TABLE I.  
Nitrogen in phenylhydrazones from diacetates and triacetates.

	Diacetates.		Triacetates.	
	%		%	
1.	..	8.33	..	7.48
2.	..	4.81	..	4.20
3.	..	8.38	..	2.64
4.	..	2.40	..	2.55
5.	..	2.12	..	1.83
6.	..	1.78	..	1.54
7.	..	1.55	..	1.33
8.	..	—	..	1.17
9.	..	—	..	1.04
10.	..	—	..	0.94
11.	..	—	..	0.86
12.	..	—	..	0.79

From Tables I. and II. it will be seen that the carbon and hydrogen content will vary to a far less extent than the nitrogen value for different members of the series contained therein. Hence if the greater number of preparations only the per

centage of nitrogen was ascertained. In a few doubtful cases the carbon and hydrogen figures were determined, and of these the hydrogen figure is the more reliable.

The experimental results are collected in Table II. for the sake of brevity. Table I. gives the nitrogen figures for the theoretically possible compounds from the di- and triacetates respectively for various values of  $n$ .

TABLE II.

Ester.*	Solvent.	Time, hrs.	M.p.	N. % found.	$n$	N. % (calc.)	Form.
Tri.F.P.	Phenol	10	229	1.02	9	1.04	R.
Tri.F.P.	Chloroform	50	239	4.16	2	4.20	R.
Tri.C.W.	Phenol	10	245	0.76	12	0.79	P.
Tri.F.P.	Toluidine	14	245	0.87	11	0.86	R.
Tri.B.C.	Toluidine	8	245	0.875	11	0.86	P.
Tri.B.C.	Pyridine	12	245	4.44	2	4.20	P.
Di.F.P.	Chloroform	12	245	1.755	6	1.78	P.
Di.F.P.	Chloroform	30	245	2.19	5	2.12	R.
Di.F.P.	Chloroform	30	245	2.22	5	2.12	R.
Tri.F.P.	Chloroform	5*	245	2.86	3	2.94	P.
Tri.B.C.	Aniline	3*	251	4.01	2	4.20	P.
Tri.B.C.	Acetic acid	8*	260	1.78	5	1.83	P.
Tri.C.W.	Acetic acid	10	262	0.90	10	0.94	P.
Tri.F.P.	Nitrobenzene	9	262	0.93	10	0.94	P.
Tri.F.P.	Aniline	3	262	2.78	3	2.84	R.
Tri.C.W.	Nitrobenzene	9	265	1.51	6	1.54	P.

\* F.P. stands for cellulose acetate from filter paper, B.C. that from bleached cotton, and C.W. that from cotton wool. Time—time in hours that mixture was actually heated.  $n$  is the value which is found to correspond with the nitrogen figure, from Table I. Form.—R=resin. P=powder. All samples marked P could not be induced to show the resinification phenomenon.

\* The solution was allowed to stand for several hours after heating.

In determinations of the melting points of these substances the temperature must be raised rapidly, as is common in the case of phenylhydrazones of simpler compounds. They all show an initial softening about 12° C. below the actual melting point, when they decompose simultaneously. A marked shrinkage in volume accompanies the initial softening.

**Acetylcellulose *p*-bromophenylhydrazones.**—It was considered that these substances would possess more marked physical properties than the corresponding phenylhydrazones. They were made in a similar manner. The final products were amorphous powders, varying from almost white to dark brown in colour. No signs of resinification were observed in any case, even when cellulose acetate derived from filter paper was employed. Initial subjection of the filter paper before acetylation to vigorous chemical purification, such as treatment with bromine or chlorine, followed by sulphurous acid, and then washing until free from impurities, did not give rise to resinous products. The melting points of these substances are sharper than those of the corresponding derivatives from the same acetylcellulose and phenylhydrazine. Further, in most cases no pronounced preliminary darkening occurs before the melting point is reached, although decomposition takes place at the melting point. In most cases these substances melt at higher temperatures than the simpler acetylcellulose phenylhydrazones. At the same time, the solubility in alcohol is less than the corresponding solubility in the simpler series. As may be expected, the results of analysis, based on the bromine content, indicate that the reaction is a progressive one, the composition of the bromophenylhydrazone produced becoming simpler on prolonged treatment before the reaction is checked. The application of heat for a short time during the reaction causes a more rapid breakdown of the complex than occurs when the mixture is allowed to stand for some time without much heating. The degradation of the complex does not proceed to such an extent as in the case of the phenylhydrazones. This is shown by com-

parison of the bromine content as found with that possible calculated from the general formula for triacetate products,  $C_{6n}H_{7n}O_{2n-1}Ac_nC_6H_4BrN_2H$ . This is indicative of a much further resolution of the cellulose acetate by phenylhydrazine than by *p*-bromophenylhydrazine. The difference of solubility of the products in alcohol also points to the same conclusion, for in general the greater the solubility the greater the degradation of the complex. Again, the melting points are higher, and in most cases are round about 287° C., and the subsequent thermal decomposition is similar to the mode in which degraded cellulose acetates decompose on heating. As the degradation of the complex increases so the melting points decrease, exactly as shown in the parallel case of the simpler hydrazones.

**Experimental.**—The method of preparation and purification was similar to that outlined for the acetylcellulose phenylhydrazones. The results obtained are shown in Table III:—

TABLE III.

Ester.*	Solvent.	Time, hrs.	m.p.	Br % found.	Br % (calc.)	$n$	Colour
DCI	Aniline	48	243	1.07	1.085	25	L.Y.
FPS	Acetic acid	22	245	3.57	3.60	7	C.B.
FPS	Acetic acid	5	265	2.66	2.62	10	R.B.
FPS	<i>o</i> -Toluidine	5	266	1.72	1.68	16	R.B.
FPS	Nitrobenzene	10	272	2.96	2.90	9	C.B.
DCI	Acetic acid	8	273	1.88	1.90	14	L.B.
FPS	Chloroform	18	278	1.01	1.01	27	L.Y.
FPI	Pyridine	17	278	2.80	2.90	9	L.B.
FPI	Phenol	4	287	0.77	0.76	36	L.B.
FPS	Nitrobenzene & acetic acid	1	287	1.08	1.085	25	L.Y.
FPI	Aniline	4	287	1.15	1.13	24	L.Y.
FPI	Acetic anhydride	16	287	1.65	1.68	16	L.Y.

\* DCI refers to cellulose acetate from bleached cotton, insoluble in acetone, and FPS to cellulose acetate from Swedish filter paper, soluble in acetone. Time—actual time of heating in hours.  $n$ —the value of the complex coefficient which is used for the purpose of calculating the theoretical Br % from the general formula already quoted. Under the heading "Colour."—L.Y.=light yellow, C.B.=chocolate brown, R.B.=reddish-brown, L.B.=light brown.

These results are indicative of the manner in which the complex cellulose is degraded by progressive resolution into simpler groups of the same empirical composition, but which contain active ketonic groups.

Chemical Laboratories,  
Cambridge.

#### THE EFFECT OF APPARATUS ERRORS ON THE ACCURACY OF VOLUMETRIC ANALYSES.

BY VERNEY STOTT, B.A., F.INST.P.

The object of the present note is to examine in detail how the results of volumetric analyses are affected by inaccuracies in the apparatus used. The errors due to other sources will be briefly indicated also. In attempting to arrive at a numerical estimate of the inaccuracy due to the errors in the apparatus involved it will be assumed that the apparatus used has the maximum errors permissible under the N.P.L. Class A tolerances,\* and that the apparatus is used without applying any corrections to the nominal capacity.

#### Errors in preparation of a solution for analysis.

The usual procedure is to dissolve a known weight of the substance to be analysed (or the whole of one

\* The tolerances allowed on Class A apparatus are given in a pamphlet, "Volumetric Tests on Scientific Glassware," published by the National Physical Laboratory in November, 1919.

constituent from a known weight of the substance) and to make up the solution to a definite volume in a measuring flask. We will assume that the flask used is inaccurate by an amount equal to the N.P.L. Class A tolerance. If the strength of the solution were to be calculated by taking the nominal capacity of the flask as its true capacity, then a percentage error would be introduced equal to the N.P.L. Class A tolerance expressed as a percentage of the nominal capacity of the flask. The Class A tolerances correspond on the average to an error of  $\pm 0.03\%$ , assuming flasks of capacity 100 c.c. to 1000 c.c. to be used.

#### Errors in preparation of standard solutions.

(A) Where a pure chemical is available a known weight may be dissolved and the solution made up to a definite volume in a measuring flask. The error in such a case would therefore be  $\pm 0.03\%$ , as above. This procedure is, however, not always possible, and other methods have to be adopted.

(a) *Standard acid solutions.*—The strength of standard acid solutions most frequently used is  $N/10$ . Such a solution may be prepared as follows: A solution which is approximately  $N/1$  is first made. Then a weighed quantity (2 g. is a convenient amount) of pure anhydrous sodium carbonate is dissolved in any convenient quantity of water, and the volume of the acid solution required to neutralise this is determined by titration. The normal acid is then diluted to  $N/10$ . Assume 2 g. of sodium carbonate to be taken, and approximately 42 c.c. of the normal acid to be required to neutralise it. If the 42 c.c. were measured from a Class A burette it may be in error by  $\pm 0.04$  c.c., i.e., by  $\pm 0.09\%$ . The dilution to  $N/10$  would probably be performed (I.) by delivering 100 c.c. of  $N/1$  acid from a pipette into a 1000 c.c. flask; (II.) by delivering 100 c.c. of  $N/1$  acid twice from a pipette into a 2000 c.c. flask; or (III.) by delivering 200 c.c. of  $N/1$  acid from a pipette into a 2000 c.c. flask.

The errors would be:—

	I.	II.	III.
Possible pipette error ..	$\pm 0.05$ c.c.	$\pm 0.10$ c.c.	$\pm 0.08$ c.c.
flask error ..	$\pm 0.20$ c.c.	$\pm 0.25$ c.c.	$\pm 0.35$ c.c.
	$\pm 0.25$ c.c.	$\pm 0.45$ c.c.	$\pm 0.43$ c.c.
i.e.	$\pm 0.02\%$	$\pm 0.02\%$	$\pm 0.02\%$

The error in dilution may hence be taken as  $\pm 0.02\%$ .

Combining this with the burette error, we obtain ( $\pm 0.09, \pm 0.02$ ), i.e.,  $\pm 0.11\%$ , as the total possible error.

(c) Decinormal oxidising solutions may be checked directly against a known weight of sodium oxalate, and in this case there would be no dilution errors. For example, 0.5 g. of sodium oxalate would be oxidised by 74.6 c.c. of  $N/10$  potassium permanganate solution. If the 74.6 c.c. were measured with a Class A 100 c.c. burette the possible error would be  $\pm 0.08$  c.c., i.e.,  $\pm 0.10\%$ . With half quantities and using a 50 c.c. burette the percentage error would be the same.

(n) *Indirectly determined standard solutions.*—Some standard solutions can only be standardised indirectly against a chemical of known and very high order of purity. The following is a method of standardising a sodium thiosulphate solution which will serve as an example. A potassium permanganate solution standardised as above (and therefore liable to an error of  $\pm 0.10\%$  due to assumed inaccuracy of apparatus) could be used to liberate iodine from an excess of potassium iodide solution. The apparatus error involved would be that of the pipette used to measure the potassium permanganate solution. If a 50 c.c. pipette were used this error would be  $\pm 0.07\%$ . The average percentage error for pipettes of capacity from 25 to 100 c.c.

would be  $0.08\%$ . The error using a 50 c.c. pipette is therefore sufficiently typical. In titrating the liberated iodine against the  $N/10$  thiosulphate about 50 c.c. of the latter would be required and be liable to an error of  $\pm 0.04$  c.c.

The total percentage error is therefore  $0.10, + 0.07, + 0.08, = \pm 0.25\%$ .

The mean of the errors in the above four cases (A—D), i.e.,  $\pm 0.12\%$ , is very close to the values for a and c, which are cases of very frequent occurrence, and therefore, although the mean has in itself no particular significance, it will be convenient to use this figure as the percentage error in standard solutions when this value is required in considering errors in the various types of analyses dealt with below.

#### Errors in analyses of Type I.

When a known weight of substance is dissolved and the whole solution titrated against a standard solution, the error of the burette used for the titration is involved. Taking 30 c.c. as an average quantity of standard solution to be used and a 50 c.c. burette as the most commonly used size, we have a possible error of  $\pm 0.04$  c.c. on 30 c.c., i.e.,  $\pm 0.13\%$ . To this must be added the error of the standard solution, viz.,  $\pm 0.12\%$ , and we thus obtain  $\pm 0.25\%$  as the total error due to inaccuracy of the apparatus used.

#### Errors in analyses of Type II.

In this case a solution is made up to a known volume and an aliquot portion taken for titration against a standard solution.

The errors in making up to known volume and taking an aliquot part vary from  $\pm 0.20\%$  with a 100 c.c. flask and a 10 c.c. pipette to  $\pm 0.05\%$  with a 2000 c.c. flask and 200 c.c. pipette. We may reasonably take  $\pm 0.10\%$  as the error arising in measuring off an aliquot portion. To this must be added the burette error in the titration and the error in the standard solution, and we thus get the total error as follows:—Pipette and flask errors,  $\pm 0.10\%$ ; burette error,  $\pm 0.13\%$ ; error in standard solution,  $\pm 0.12\%$ ; total,  $\pm 0.35\%$ .

#### Errors in analyses of Type III.\*

This may be described as the excess method, and involves the use of two standard solutions.

Suppose A and B to be the two standard solutions and C the solution whose strength is required.

To take a particular case, suppose 50 c.c. of A is equivalent to 50 c.c. of B, and that when 50 c.c. of B is added to 50 c.c. of C, 30 c.c. of A is required to complete the neutralisation of B. Thus 20 c.c. of B is equivalent to 50 c.c. of C.

The errors involved are as follows:—The strength of the solution A added from the burette in the first case to 50 c.c. of B, and in the second case to 50 c.c. of B and 50 c.c. of C need not be known. The solution A simply serves to give the ratio 20 c.c./50 c.c. obtained from the burette readings. This ratio may be in error by 0.2%. The error in taking an aliquot portion of C would also be involved, and the error in the assumed strength of B, and in measuring the amount of B taken from the test. The total errors would therefore be:—Error in ratio of burette readings, 0.2%; in making up C and taking a portion, 0.1%; in measuring B (average pipette error), 0.08%; in standard B, 0.12%; total, 0.50%.

If C were a solution of the whole of a constituent from a weighed portion of a substance, e.g., a solution of the phosphorus from a weighed quantity of steel, then the error in taking an aliquot portion would not arise and the above value would reduce to 0.40%.

\* The bismuthate method of determining manganese in steel is an example of Type III.

*Summary of errors in typical cases.*

The errors in the cases considered are summarised in the table below:—

	Type I.	Type II.	Type III.
Standard in error by $\pm 0.03\%$	$\pm 0.16\%$	$\pm 0.26\%$	$\pm 0.41\%$
" " " $\pm 0.11\%$	$\pm 0.24\%$	$\pm 0.34\%$	$\pm 0.49\%$
" " " $\pm 0.26\%$	$\pm 0.39\%$	$\pm 0.49\%$	$\pm 0.64\%$

The mean of the values given in the above table is  $\pm 0.38\%$ . The value  $\pm 0.4\%$  may therefore be taken as an average value for the greatest error which may arise in a volumetric analysis due solely to inaccuracies in the apparatus used, it being assumed that the apparatus used has the maximum errors allowed under the N.P.L. Class A tolerances.

*Significance of above error.*

The figure  $\pm 0.4\%$  represents the possible range of the total error due to inaccuracies of the apparatus used, and not the probable error in any particular case. The figure assumes all errors to be cumulative, whereas they may be compensating, i.e., the figure relates to the worst case which may arise.

The probability of the errors being cumulative as regards sign is  $1/2^n$  where  $n$  is the number of pieces of apparatus used. For example, using a flask, pipette, and burette, the probability of the errors being cumulative is  $1/8$ . Hence, unless more than five or six different pieces of apparatus are involved, the probability of the errors being cumulative as regards sign cannot be considered small.

The probability of the errors in the apparatus having the maximum value allowed under the Class A tolerances cannot be assigned a definite value. It should be noted, however, that the Class A tolerances represent a high standard of manufacture, and that in ordinary commercial grade apparatus larger errors than the maximum allowed under the above tolerances are quite likely to occur.

Another important point is the significance which the above error would have in the final form in which the results of the analysis are expressed. In a large number of instances the object of a volumetric analysis is to determine the percentage of one constituent of the substance under examination. If there is  $x\%$  of the constituent present, and the error in its determination due to the cause considered above is  $n\%$  of that constituent, then the error expressed as a percentage of the whole substance is  $nx/100\%$ . Thus on a  $10\%$  constituent the average maximum value of  $0.4\%$  derived above for the error in the volumetric determination would be  $0.04\%$  when expressed as a percentage of the whole substance.

The same matter may be viewed in a slightly different manner as follows:—If a  $10\%$  constituent is determined volumetrically and the result is required correct to the first decimal place, then the total errors from all causes should not exceed  $\pm 0.05\%$  of the whole substance, i.e.,  $0.5\%$  of the  $10\%$  constituent.

We have seen that the errors due simply to the apparatus used being inaccurate to the extent of the N.P.L. Class A tolerance may amount to  $0.4\%$  of the constituent, taking an average of the maximum values.

It is clear, therefore, that when a constituent is to be determined to an accuracy corresponding to  $0.5\%$  or even to  $1\%$  of the percentage of the constituent present, the errors of the apparatus used are a factor requiring careful consideration.

*Other sources of error.*

(1) *Errors in weighing.*—These should be negligible if proper precautions are taken.

(2) *Errors of manipulation.*—Such are: (a) incorrect setting of liquid meniscus on graduation marks of pipettes and flasks or incorrect reading of position of liquid surface in burette; (b) not strictly adhering to standard methods of delivering liquid from pipettes and burettes; (c) loss of liquid by splashing etc.

The errors under (a) may be placed at approximately from  $1/5$ th to  $1/10$ th of the N.P.L. Class A tolerances. The errors under (b) are not likely to be more than  $1/4$ th the N.P.L. Class A tolerances when standard apparatus is used. The errors under (c) should, of course, be negligible.

(3) *Errors due to uncertainty of end-point.*—This varies with the method of observing the end-point. With ordinary indicators as generally used, the value  $0.03$  c.c. should cover this source of error.

(4) *Errors due to temperature differences.*—If standard solutions are made up within one or two degrees of  $15^\circ\text{C}$ ., and solutions are allowed to assume room temperature before titration, no serious errors need arise from this cause.

(5) *Chemical errors.*—Under this heading come such sources of errors as the partial oxidation of ferrous iron to the ferric state before the addition of the standard oxidising solution, the incomplete separation of closely allied elements, etc. Such sources of error are of a different nature from those dealt with above, and relating as they do to particular methods do not properly arise in considering errors common to volumetric analyses in general.

(6) *Uncertainties in the accepted values of the atomic weights.*—These again are of a particular nature beyond the scope of the present paper.

*General conclusions.*

Excluding such causes as (5) and (6) just given, it is interesting to frame some estimate of the probable percentage error arising from the other causes just enumerated. No exact figure of general application can be given. In the opinion of the author a minimum estimate would be  $0.4\%$ . To assign a maximum figure is more difficult. Twice or three times the minimum would be a fairly reasonable estimate, but the question is one on which it is impossible to be precise.

Finally it has been shown that  $0.4\%$  may be taken as a fairly representative average of the maximum errors which may arise in volumetric analysis due solely to apparatus errors when using apparatus accurate within the N.P.L. Class A tolerances, but used without applying corrections.

A more debatable conclusion is that a minimum estimate of the errors from other sources is also approximately  $0.4\%$ .

The National Physical Laboratory.  
January, 1921.

## THE EROSION OF BRONZE PROPELLERS.

### CONTRIBUTION TO DISCUSSION.

Mr. WILLIAM RAMSAY (of Messrs. Cammell Laird and Co., Ltd., Birkenhead) wrote that Dr. Silberrad's paper (J.L. 1920, 387) was of special interest to him as he had been engaged on the same work for some twenty years. Although Dr. Silberrad and the "Propeller Committee" appeared to be satisfied that the cause of the defect has been definitely settled as a purely mechanical action, others were not so convinced that the correct explanation had been given. The "Propeller Committee" had consisted of Sir Charles Parsons and a number of engineers and naval officers who, although of very high rank in their own professions, were hardly fitted to decide on a

metallurgical or chemical question. The only other member who could deal with the matter in an authoritative manner had been Prof. H. C. H. Carpenter. On the strength of a number of somewhat unconvincing experiments the Committee had decided that the defect was due to water-hammer action, the metal being broken away by impact and not by frictional wear or erosion. However weak their experimental evidence had been in favour of water-hammer action, it had certainly proved the erosion theory to be a fallacy.

Dr. Silberrad's claim that his alloy had overcome the defect was not, in his opinion, fully proved, his (the writer's) own experience being that such propellers were no better than those made of the old manganese brass. In his article on the subject published in "Engineering," May 24, 1912, several photographs of the defect appeared, all of which had been taken from blades of the Silberrad alloy, and a large stock of such propellers had been discarded as unsafe. It was not safe to rely on the presence or absence of copper concentration on the corroded surfaces for evidence of chemical action. In the conditions under which high-speed propellers worked such removal of zinc or copper concentration was impossible. Some recent work of his own had shown that this phenomenon was entirely due to redeposition of copper from the corrosion product, and if the latter were swept away as formed no redeposition of copper took place.

He had carried out many erosion tests on similar lines to those of Dr. Silberrad, but using a high-pressure jet of Mersey water. In no case had he been able to obtain more than a slight roughening of the disturbed surface layer of metal; the extent of the action appeared to depend very largely on the manner in which the surface of the metal was prepared. If, however, the jet was made the negative and the metal the positive of a circuit supplying a small current such as might exist between stressed and unstressed metal, interesting results were obtained. At low water pressures the metal became coated with corrosion product, and the current fell off almost to nothing in a short time. At a higher pressure the corrosion product was washed away, and the metal (brass) was seen to be coated with redeposited copper. At still higher pressure this copper was removed and the corroded surface became bright and of the natural colour of the metal. Further, the loss in weight was invariably greater than that demanded by the current supplied; in one case the excess corrosion amounted to over 800%, though as a rule it was from 50% to 200%. Specimens corroded in the above manner reproduced in miniature all the features of a corroded propeller blade. No distinction could be drawn between  $\alpha$ -brass and  $\beta$ -brass or one containing both phases. Pure metals behaved similarly, and, as one might expect, aluminium and zinc appeared to suffer most. Further investigation into the matter was required, but it appeared

to him that such combined action might be sufficient to account for propeller deterioration.

Dr. Silberrad's tests on stressed and unstressed brass showed a small but consistently greater loss on the stressed material. A high-speed propeller blade, however, instead of being stressed four times per minute, probably suffered alternating stresses of the order of 800 per minute and possibly a much higher frequency, and had Dr. Silberrad or the "Propeller Committee" approached these conditions in their experiments it was possible that their results and conclusions might have been very different.

The practice of backing metal with soft solder for sectioning for micro-examination was, in his (the writer's) experience, open to objection, and he was surprised that Dr. Silberrad had not found many more distorted  $\alpha$ -crystals. There was a growing suspicion among micro-metallurgists that even such a mild process as polishing a comparatively soft metal such as brass distorted the surface layer of crystals to some slight extent even under the amorphous polish layer. This was sometimes seen in an exaggerated form at the filed or sawn edges of a section. Embedding in solder, Wood's alloy, or in electro-deposited copper did not entirely prevent this, and crystals were frequently torn completely out at the junction of the two metals. The presence of distorted crystals at the edge of a fracture or a surface such as Dr. Silberrad had examined should therefore be accepted with certain reservations.

When the turbine had first been used for marine propulsion, owing to the high shaft speed and other considerations, it had been considered necessary to cut down blade weight to the lowest limit, the result being that at top speeds (about 800 revs. per minute) considerable "whipping" of the blade had ensued. Whether this was the cause of the trouble or not was still doubtful, but the use of stiffer blades had reduced the defect and, he understood, eliminated it in some cases. The present tendency to increase efficiency by reducing shaft speed by the intermediary of single or double reduction gear and stiffer propellers had practically removed the trouble, and to this extent The Manganese Bronze Co. and other equally good propeller founders might fairly claim that their alloys were erosion-proof.

The references to Sir William Ramsay called for some explanation. Sir William admittedly had not been an authority on the subject, and had intervened simply because someone had attributed the present writer's views on the subject to himself owing to their names being the same. The opinions which he had volunteered as to the universal use and success of the new alloy had been formed entirely on Dr. Silberrad's statements and not from first-hand knowledge of the subject. The continued use of Sir William Ramsay's name in that connexion therefore appeared to be unnecessary.



# Joint Meeting of the Society of Chemical Industry and of the Institution of Mechanical Engineers.

Held at the Institution of Mechanical Engineers  
on March 4, 1921.

## DEGASSING AND PURIFICATION OF BOILER FEED-WATER.\*

BY PAUL KESTNER.

(Abstract.)

The problem of feeding boilers with water in such a way that they will run for long periods under steam without scale or corrosion has not been completely solved up to the present. The old limo and soda purifying apparatus has been improved and reduced in size, and heat has been applied to accelerate the process, but even with these improvements some kinds of water have proved refractory, scale being formed, or in some cases a concentrated solution of soda being produced in the boiler. A process has, however, been devised which prevents the formation of mud and incrustation completely. It consists of continuous blowing-down, used in conjunction with the hot purification process. Hot purification processes result in increased speed of reaction, more rapid evolution of dissolved gases, diminished viscosity of the water (resulting in more rapid settling of the precipitate), decomposition of alkaline-earth bicarbonates, and decreased solubility of calcium sulphate.

The usual method adopted to remove the sediments from boilers is to blow off the water periodically, but this necessarily involves loss of heat. To be effective, blowing off must take place to the extent of a large actual quantity of water, though the amount is small in proportion to the total contents of the boiler. In the continuous blowing down process, instead of sending the blow-off direct into the drain a relatively small quantity is removed from the boiler continuously at its lowest point and is passed through a feed-water heater of the direct contact type where it gives up its heat, the mud being deposited in a reservoir below the heater; the water in cooling loses a certain proportion of the salts which had been retained in solution and is then mixed with the feed-water and returned to the boiler. For example, the boiler may be blown down to the extent of 20% of its contents in an hour, i.e., its contents are renewed every five hours; this ensures the elimination of all mud that has been produced before it has had time to accumulate. When the feed-water is purified by means of sodium carbonate, this reacts with the calcium bicarbonate, forming sodium bicarbonate, which is decomposed in the boiler into carbonate; a certain amount of sodium hydroxide is also formed in the boiler as a result of the hydrolysis of the sodium carbonate. In the continuous blowing down process, therefore, the feed-water is continuously supplied with a solution of sodium hydroxide and carbonate. If the quantity of water removed from the boiler in a given time is calculated so that it contains the quantity of soda necessary for the purification of the water entering the boiler in that time, it will not be necessary to add any sodium carbonate to remove the temporary hardness of the water.

The apparatus illustrated in Fig. 1 has been devised to use a blow-down on the described

principle. It consists of a steam separator, S, which the blow-down enters in the form of a mixture of steam and water. The liquid from the blow-down falls into a central reservoir, P, in which it gives up its heat to the water surrounding it in the annular chamber, B, into which it is decanted. The steam from the blow-down rises in the reheater, R, in which it heats the feed-water, which thus loses a part of its bicarbonate. The heated water falls through the tube, T, and mixes with the blow-down as it leaves the central chamber, and then rises in the annular chamber, B, in which precipitation and settling take place. The purified water then passes through the filter and enters the boiler. The soda is distributed in the apparatus by means of a water meter, J, which admits it in proportion to the new water (make-up feed). The valves, V and V<sub>1</sub>, allow the mud to be separated.

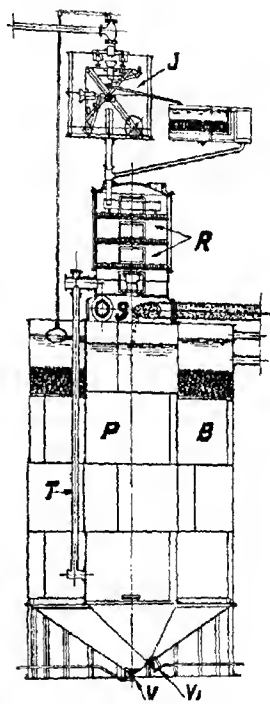


FIG. 1.

The blow-down must be arranged at the lowest possible level on the boiler without risk of emptying it. A simple arrangement (Fig. 2) is recommended by which, when the water of the boiler falls below a certain level, the siphon action of the blow-off is broken. At this moment steam leaves by the inlet and gives warning by sounding a whistle. In any case it is impossible to empty the boiler by the continuous blow-down. On the pipe of the continuous blow-down and the outlet from the boiler a reducing valve is placed, which reduces the pressure of the blow-down from that of the boiler to a pressure of 1-2 kg. per sq. cm. (15-30 lb. per sq. in.) before it enters the purifier. A meter, fitted with a regulating valve, is placed at the outlet of the boiler.

\* The full paper will appear in an early issue of the Proceedings of the Institution of Mechanical Engineers.

The proportions of the blow-down are calculated according to the temperature to be maintained in the purifier, and the rate of blow-down depends on the working pressure of the boiler. The temperature at which purification is effected should not be less than 70° C.

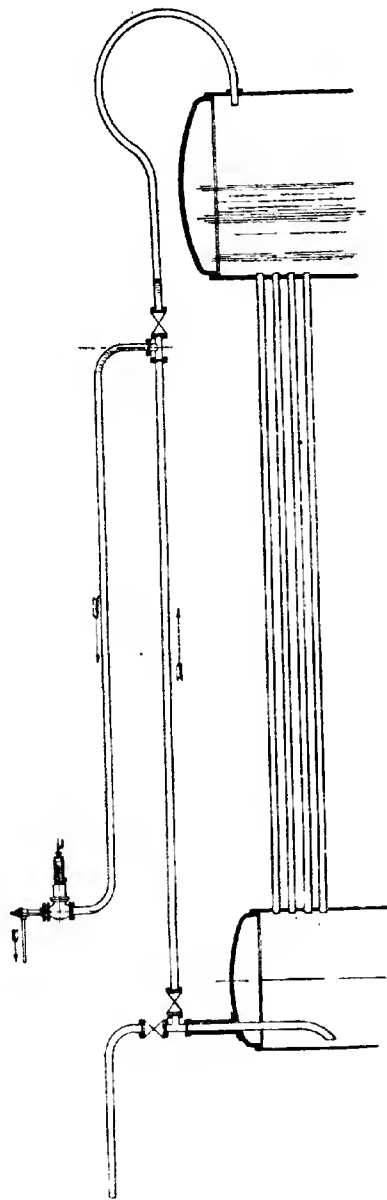


FIG. 2.

In central stations using distilled water, small amounts of soluble salts are carried over by entrainment, so that the salts gradually become concentrated in the water. Boilers working with distilled water therefore require to be blown down occasionally, the amount removed being usually 1–2 %, which is replaced by distilled water. Even when an evaporator is used the continuous blowing-down method can be adopted; a certain amount of

saline or impure water can be drawn from the boilers and passed into the evaporator, which returns it to the boiler after it has been freed from the greater portion of its salts. By means of continuous blowing-down, in conjunction with degassing, a complete closed water circuit can be established in a central station using distilled water.

In the case of boilers using distilled water corrosion can only be produced by oxygen dissolved in the feed-water and set free in the boiler, and the effect is greater with intermittent working.

In the case of boilers using purified water other factors have to be considered before coming to a conclusion. The corrosive effect of saline solutions increases in proportion to the concentration up to a critical point at which it attains a maximum; the effect then decreases rapidly as the concentration increases. In practice, after a few hours, a boiler using purified water will have already attained a concentration fully one hundred times the critical amount, so that corrosion cannot be attributed to this cause. Electrical action may have some influence on corrosion, but it is not of great importance. Electrolytic dissociation occurs much more easily in a dilute than in a concentrated solution, and is of little importance in connexion with corrosion.

Hydrolysis may have more important effects, particularly in the case of magnesium chloride, which gives rise to the production of hydrochloric acid. Fortunately, this salt only occurs occasionally, and its effects are sufficiently well known for special care to be given where it is present. Carbon dioxide alone has only feeble action on iron, but in presence of oxygen the metal is attacked much more rapidly. Oxygen comes into play still further when the water contains salts capable of combining with oxygen and forming unstable oxygen compounds which can part with their oxygen to form other oxides.

Thus it appears that it is the solution of atmospheric oxygen in water that is the chief cause of corrosion, whether the feed is distilled water or purified water. This is supported by the fact that corrosion is often more serious in boilers using distilled water, in which dissolved oxygen can be the only agent, than in boilers using purified water. The remedy is simple, namely, the elimination of the dissolved oxygen.

The solubility of air in water follows Henry's law. At ordinary temperatures and pressures 18 c.c. of nitrogen and 35 c.c. of oxygen form saturated solutions with 1 litre of water. The quantities of each dissolved from air are therefore: Nitrogen:  $0.8 \times 18 = 14.4$  c.c.; oxygen:  $0.2 \times 35 = 7.0$  c.c. The air dissolved in water will therefore contain, roughly, 33% of oxygen. Laboratory tests have shown that the quantity of oxygen dissolved in water varies slightly with the nature of the water, and soft water absorbs more than fresh water, and soft water more than hard water. Distilled water absorbs 7.8 c.c. per litre at 20° C., 2.1 c.c. at 100° C. The water supply of Paris contains 6.06 c.c. per litre at 20° C., 4.3 at 55° C., and 2 c.c. at 100° C.

When water is heated supersaturation occurs. No gas is evolved up to a temperature of 75° C. Air is only driven off continuously when the water is agitated continuously from the commencement of heating. When water that has been boiled is cooled the inverse takes place; a litre of water absorbs, for instance, 2.3 c.c. at 44° C. and 3 c.c. at 20° C. If the water is agitated in air the false state of equilibrium is disturbed, and the water becomes saturated up to 6 c.c. of oxygen. The water resulting from the condensation of steam in the air contains 4 c.c. of oxygen per litre at 80° C. The same quantity of water of condensation exposing only a small surface to the air contains only 3 c.c. of oxygen per litre at the same temperature.

The determinations of dissolved oxygen have been made by the following modification of Winkler's method: The water is introduced into a 2-litre flat-bottomed flask through a tube dipping into a small quantity of paraffin; 1 g. of manganese sulphate is added and the flask is immediately closed by a rubber stopper fitted with a funnel having a stop-cock and a glass tube connected by rubber tube to another glass tube of larger diameter, carried on a stand. It is possible, by raising or lowering the tube, to change the pressure in the flask and to cause liquid contained in the funnel to be drawn into the flask. When the manganese sulphate is dissolved, 5 c.c. of sodium hydroxide solution is introduced through the funnel and the flask is shaken. After a quarter of an hour the absorption of oxygen by the manganous hydroxide is complete. Two c.c. of a 50% solution of potassium iodide (free from iodate) is introduced through the tube fitted with the funnel, followed by 20 c.c. of concentrated hydrochloric acid. The flask is shaken until the precipitate has dissolved and the amount of iodine liberated is determined by

These can only have the effect of preventing super-saturation at the particular temperature and pressure. Even if the temperature of the water is raised to 100° C. the solubility of the gases cannot be neglected. Heating, however, produces a great improvement in the condition of the water entering the boiler. A vacuum would give better results still if it could be used at a very high temperature, but the vacuum cannot be very high, otherwise the water would boil. A large German company which makes an apparatus using these three methods simultaneously states that water treated by this process still contains some milligrams of oxygen that cannot be removed by a very high vacuum and by reheating. Nevertheless, the physical removal of gases diminishes the percentage of oxygen in the feed-water and retards the corrosion.

All chemical methods of removal of gases in actual use consist in passing the water over iron. If the iron has a sufficiently large surface, if it is of a kind readily oxidised, and if the water is circulated sufficiently slowly, all the oxygen can be eliminated.

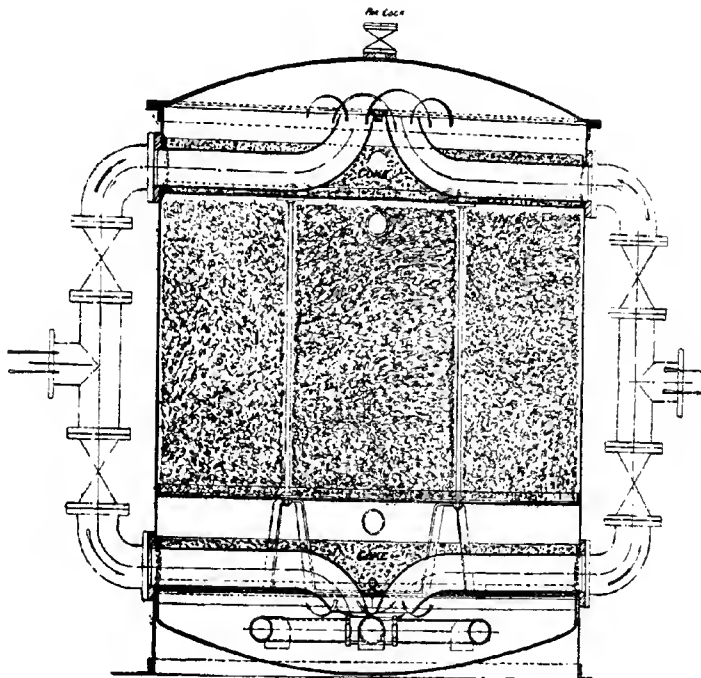


FIG. 3.

titration with standard sodium thiosulphate, preferably of such strength that 1 c.c. = 1 c.c. of oxygen.

The removal of the oxygen dissolved in water may be effected either by physical methods or by chemical methods, or by the two combined. The principal physical methods are agitation of the water, heating, and reduction of pressure. These three methods may be used either separately, or in the majority of cases, in combination. Of the chemical methods the only one to be considered is the action of highly oxidisable iron, which absorbs the oxygen from the water before entering the boiler.

Agitation of the water is used in various appliances in the form of fine spraying or circulation through channels of varying section, or by passing the current of water over an uneven surface.

Laboratory experiments have shown that manganese accelerates the oxidation of iron, and a suitable manganese cast-iron for the purpose has been found.

In commercial appliances, when the coating of oxide becomes very thick, the removal of the oxygen may become considerably slower, but under the most unfavourable conditions water leaving such apparatus is found to contain less than 0.5 c.c. of oxygen per litre.

To avoid the retardation of the oxidising process by the deposit on the iron the Kestner Co. has designed an apparatus based on the observation that if the oxidised iron is allowed to remain at rest for a day the red deposit will become greenish-black owing to the reduction of the ferric hydroxide by the iron. This reaction takes place in the middle

of the reducing agent—that is, in the presence of iron and in the absence of oxygen. The product is easily removed from the iron by washing, and, moreover, is capable of further absorbing oxygen to form ferric hydroxide.

The Kestner apparatus (Fig. 3) consists of a column of iron turnings placed between two filters with an intake and outlet for the water at each end of the apparatus, in such manner that the direction of flow of the water can be reversed at regular intervals, and an arrangement for allowing steam to be admitted at the base for cleaning.

The column of turnings is of such quantity that one-half only is in action, and that the gases are removed when the water has reached half the height. At the end of the day the direction of flow is reversed. The consumption of iron turnings varies from 1 to 3 g. per ch. m. of water.

Whatever may be the method employed for removing the gases, it involves a circuit of water closed from atmospheric contact if it is to give a large output. It is therefore advisable that the tanks in which the water is retained should be as well enclosed as possible, and that on the whole pipe system there should be a small positive pressure.

In such a system the only place at which the entry of air should be possible is the condenser in which the vacuum is produced. The tightness of the condenser should therefore be very carefully tested, and the water should pass through the gas remover immediately before entering the boiler.

The study of continuous blowing down and of degassing has led to the conception of a complete circulation system for a central station using distilled water, in which one boiler evaporates the purified water coming from the purifier and supplies steam that passes with that generated by the other steam boilers to the steam main. The steam passes through turbines, is condensed, and re-enters the boilers as distilled water after it has passed through the degassing apparatus. All the boilers are blown down, and the amounts blown down are passed collectively through the purifier. The purified water is returned to the distilling boiler after it has passed through a special degassing appliance.

The boiler containing the water to be purified thus furnishes the supply to the boilers working with distilled water in the form of steam. It is only necessary to provide an overflow reservoir to enable any excess of purified water to be re-introduced into the circuit if such excess should be produced. All the boilers can be fed either with purified water or with distilled water, which enables any desired boiler to be used for distilling.

## Birmingham Section.

Meeting held at the University, Birmingham, on Thursday, January 27, 1921.

DR. H. W. BROWNSDON IN THE CHAIR.

### THE PREPARATION OF ZIRCONIA FROM BRAZILIAN ORE AND A NEW METHOD OF DETERMINATION.

BY E. C. ROSSITER, A.C.G.I., F.I.C., AND P. H. SANDERS, A.I.C.

\* In the extraction of zirconia in a pure state from its ores one of the chief difficulties is the complete separation from iron and alumina. This paper describes a simple method for this separation and one which has been found to give practically quantitative results.

The method is based on the precipitation of the zirconia as a basic sulphate in which the ratio of zirconia to sulphuric acid is expressed by the formula  $5\text{ZrO}_2 \cdot 2\text{SO}_3$ . A similar method has been proposed by K. Leuchs (G.P. 285,344, 1915) in which a basic sulphate of the formula  $7\text{ZrO}_2 \cdot 3\text{SO}_3 \cdot 14\text{H}_2\text{O}$  is precipitated; Leuchs has modified this method (G.P. 295,246, 1916) so as to obtain a crystalline basic sulphate of the formula  $3\text{ZrO}_2 \cdot 2\text{SO}_3 \cdot 6\text{H}_2\text{O}$ . Both these products are claimed to be free from iron, but the yield is not stated. A similar method is described by the Pennsylvania Salt Manufacturing Co. (E.P. 153,113) in which a crystalline basic sulphate of the composition  $5\text{ZrO}_2 \cdot 3\text{SO}_3 \cdot 13\text{H}_2\text{O}$  is obtained by adding the requisite proportion of sulphuric acid to a solution of the oxychloride; this basic sulphate is claimed to be substantially free from iron, titanium, and silica, but again the yield is not stated.

The ore used in the present investigation was from Brazil; it was ground fine in a porcelain ball mill before being used; during the grinding it was found that about 50% was much softer than the remainder and this was separated; on further grinding a small residue amounting to 0.8% remained unground. These three portions were analysed separately and gave the following figures:—

#### Analysis of the ore.

	Softer portion.	Hard portion.	Very hard residue.
ZrO <sub>2</sub> .. ..	65.00	75.10	74.30
SiO <sub>2</sub> .. ..	24.48	18.50	17.28
Fe <sub>2</sub> O <sub>3</sub> .. ..	4.90	1.75	4.80
Al <sub>2</sub> O <sub>3</sub> .. ..	4.30	4.05	2.70
Loss on ignition ..	1.25	0.50	0.50
	99.03	99.90	99.58

The ore also contained traces of titanium and manganese and gave a slight indication of thorium.

When this ore is extracted with hydrochloric acid for 3 days at room temperature only 41% of the iron and alumina is dissolved. When extracted at 100° with hydrochloric acid only 45% of the iron and alumina is dissolved and 0.25% of the zirconia.

#### Decomposition of the ore.

Two methods of decomposing have been examined, namely fusion with caustic soda and fusion with acid sodium sulphate (nitro-cake).

Fusion of the ore, previously ground to flour fineness, with its own weight of caustic soda at a final temperature of about 600°C. yields a product which, after extraction with hot water, is soluble in hydrochloric acid to the extent of 80% of the ore. Fusion of the ore with four times its weight of nitre-cake yields a product which on extraction with water yields a solution containing 50% of the ore.

Since, as will appear later, it is necessary to obtain the zirconia in solution as oxychloride in order to separate it completely from the iron and alumina, fusion with caustic soda is preferred. 50 g. of the zirconia ore is mixed with 50 g. of caustic soda and fused in an iron crucible; foaming occurs and the mixture is continually stirred until it becomes a granular powder; the temperature is then raised to just visible redness. The fusion takes 2 hrs. The product is thrown hot into 1½ l. of water, filtered, and the residue washed free from alkali. The insoluble residue is then treated with hydrochloric acid and the mixture evaporated to dryness on the water bath. The product is then extracted with hot water and filtered. The insoluble residue after the fusion process amounts to 19–20% of the ore used, and contains 61.55% ZrO<sub>2</sub>, 29.32% SiO<sub>2</sub>, 1.10% Fe<sub>2</sub>O<sub>3</sub>, 1.80% Al<sub>2</sub>O<sub>3</sub>, 5.90% loss

on ignition. The solution contains zirconium oxychloride, and iron, alumina, titanium, manganese, etc.

#### *Precipitation of the zirconia.*

The solution of the chlorides is diluted to 1½ l., treated with a slight excess of sulphurous acid over that required to reduce the iron salts (about 100 c.c. of saturated SO<sub>2</sub>), and heated to boiling. N/1 Sulphuric acid is then gradually added in amount equal to that theoretically required to replace the chlorine in the oxychloride or until the mixture becomes quite thick; after a short time the whole of the zirconia is deposited as basic sulphate. This precipitate is easy to filter, and settles rapidly; it is, however, very bulky, and we prefer to wash it by decantation. Five washings by decantation in a glass cylinder of 6 l. capacity reduce the soluble impurities to 0.3% of the amount originally present. The precipitate is then drained on a filter. The wet precipitate gives only the faintest indication for iron when tested with thiocyanate, and therefore contains less than 1 part in 100,000.

To obtain zirconia from the basic sulphate it may be either dried and ignited, or suspended in water, treated with alkali, and the hydroxide washed, dried, and ignited. The zirconia obtained contains 98–99% ZrO<sub>2</sub>, the remainder being silica and a small quantity of alumina. The above precipitation as basic sulphate is practically quantitative, and affords a ready means of determining zirconia in the presence of iron and alumina.

Zirconia is precipitated from the boiling solution of its oxychloride by sulphuric acid alone, and also by sulphurous acid alone, but the precipitates are usually very difficult to filter and wash, and in no case have we been able to obtain a complete precipitation under these conditions. The exact action of the sulphurous acid has not been determined; it was added in the first instance to prevent the precipitation of iron, which always comes down in small quantity with the zirconia in the absence of sulphurous acid.

#### *Properties of the basic zirconium sulphate.*

The basic zirconium sulphate when dried at room temperature or at 100° C. has the appearance and colour of clear amber. The sample dried at room temperature contained 51.92% ZrO<sub>2</sub>, 13.53% SO<sub>3</sub>, and 34.53% H<sub>2</sub>O, corresponding with the formula 5ZrO<sub>2</sub>·2SO<sub>3</sub>·22.6H<sub>2</sub>O. The sample dried at 100° contained 65.36% ZrO<sub>2</sub>, 17.03% SO<sub>3</sub>, and 17.80% H<sub>2</sub>O, corresponding with the formula 5ZrO<sub>2</sub>·2SO<sub>3</sub>·9.3H<sub>2</sub>O. It loses water progressively as the temperature of drying is raised. After drying at 100° it is easily soluble in concentrated sulphuric acid, slightly soluble in hydrochloric acid, and completely decomposed by prolonged boiling with sodium carbonate or ammonia. When ignited it blackens and is decomposed to the oxide. Before drying it is easily and completely soluble in hydrochloric acid, and when this solution is evaporated on the water-bath crystals of zirconium oxychloride are obtained, the mother liquid containing zirconium sulphate.

The hydroxide prepared by suspending the undried basic sulphate in ammonia is easily soluble in hydrochloric acid, and this solution when concentrated on the water-bath and allowed to crystallise yields the oxychloride ZrOCl<sub>2</sub>·8H<sub>2</sub>O, crystallising in slender needles. An analysis of this oxychloride gave 38.20% ZrO<sub>2</sub>, 21.87% Cl, whereas the formula requires 38.12% ZrO<sub>2</sub>, 22.12% Cl.

The basic zirconium sulphate described above has all the properties of the 5:2 basic zirconium sulphate described by Rodd (Chem. Soc. Trans., 1917, 111, 396), except that the hydrate when dissolved in hydrochloric acid yields the normal oxychloride. No trace of Rodd's 5:4 basic zirconium chloride was observed. Rodd also describes a basic sulphate obtained by adding sulphuric acid to a

dilute solution of zirconium oxychloride previously boiled for 6 hrs.; this basic sulphate on analysis gave figures corresponding with those of the 5:2 basic sulphate, but after drying the salt was insoluble in sulphuric acid and was unattacked by ammonia, and is therefore different from the 5:2 basic sulphate described above, and also from Rodd's 5:2 basic sulphate.

Attempts to make Rodd's 5:4 basic zirconium chloride according to the method described by him always resulted in obtaining the ordinary zirconium oxychloride ZrOCl<sub>2</sub>·8H<sub>2</sub>O. Nor were we able to prepare a 5:2 basic sulphate which was different from that described above.

#### *The determination of zirconia in the presence of iron and alumina.*

Since the precipitation as basic sulphate described above was apparently quantitative, this reaction was examined to determine under what conditions it could be used in controlling the process of manufacture. Tests on a solution of zirconium oxychloride of known strength indicated that in order to obtain quantitative results, 7 c.c. of sulphurous acid solution and 2 c.c. of N/1 sulphuric acid should be used for each 0.2 g. of ZrO<sub>2</sub>, and that the solution should be diluted to at least 150 c.c. for each 2 c.c. of sulphuric acid. The difficulty to be overcome in the presence of iron is the quantity of sulphuric acid formed in the reduction of the ferric salts to ferrous by the sulphur dioxide. Good results were obtained in the presence of iron in the proportion of 1 part of Fe<sub>2</sub>O<sub>3</sub> to 3 parts ZrO<sub>2</sub> by proceeding as above and, by modifying the method, complete separation from quantities of iron oxide up to 3½ times that of the zirconia. Attempts were made by neutralising the solution with ammonia after the reduction, but the results were high on account of some iron being precipitated along with the zirconium basic sulphate.

By the following method, however, good results were obtained. The solution used contained 0.1906 g. ZrO<sub>2</sub> and ferric chloride equivalent to 0.7 g. Fe<sub>2</sub>O<sub>3</sub>, diluted to 150 c.c.; 25 c.c. of sulphur dioxide solution was added and the mixture boiled until the iron was reduced. It was then neutralised with ammonia, 10 c.c. of sulphur dioxide solution and 2 c.c. of N/1 sulphuric acid added, and the liquid again boiled. The precipitated basic sulphate was filtered off, washed, and redissolved in hydrochloric acid. This solution was evaporated just to dryness to remove excess of acid, the residue dissolved in water, and the zirconia again precipitated with 10 c.c. of sulphur dioxide solution and 2 c.c. of N/1 sulphuric acid. The ignited precipitate weighed 0.1900 g. and was quite white (error, 0.31%).

With much larger proportions of iron the method does not give good results since the basic sulphate is somewhat soluble in the ammonium sulphate and chloride formed during the neutralisation.

Research Laboratories,

The British Cyanides Co., Ltd.,  
Oldbury.

#### DISCUSSION.

Mr. BRADFORD said that the method appeared to have some points of resemblance with the patented method of Rodd. The difficulty of separating zirconia from baddelyite and other ores and obtaining a pure sample was exceptionally great. He had devised a method quite different from that of the National Physical Laboratory in which ore was opened up by means of hydrofluoric acid; the extraction was carried out on an industrial scale, and he obtained zirconia which was dead white. He was interested in the quantitative separation of titanium from zirconia, and would welcome

information on this point. Experiments showed that the presence of iron, alumina, titanium, etc., did not interfere with the quantitative determination of zirconia, if the latter was determined by means of "cupferron" as elaborated by Thornton and Haydon.

Mr. F. H. ALCOCK suggested that an effective method for opening up refractory ore was by fusing with calcium oxide or with potassium hydrogen sulphate.

The CHAIRMAN suggested the opening up by fusion with acid sulphates as leading to a possible simple analytical method when insoluble sulphates were involved. In dealing with rich lead alloys, especially those containing a few per cent. of antimony, he had found direct fusion with potassium acid sulphate and subsequent treatment of the melt with water a rapid means of obtaining the insoluble sulphate, and, indirectly, especially convenient for the titration of antimony in the filtrate.

Mr. ROSSITER, in reply, said the process was very similar to that described by Rodd, the chief difference being in the use of sulphurous acid. Rodd, in his paper, pointed out that the precipitation of the basic sulphate depended upon the previous history of the solution of zirconia, but the previous history of his solution was not given. A very complete account of analytical methods for opening up and analysing zirconia ores had been given by Lundell and Knowles (J. Amer. Chem. Soc., 1920, 42, 1439). The separation of zirconium from titanium was not complete by the method described. Sodium salts were used in preference to potassium salts in opening up the ore because zirconia formed difficultly soluble salts with potassium salts. The solution obtained by fusing the ore with acid sodium sulphate and lixiviating the product would on boiling give a basic zirconium sulphate precipitate, providing it was nearly neutral, but this precipitate would most probably be difficult to filter and wash, it would not settle, and would not be free from iron. It was difficult to obtain anything like a quantitative separation of zirconia as basic sulphate from an acid solution; that was the reason why, after the ore had been opened up by caustic soda and the part insoluble in water extracted with hydrochloric acid, it was necessary to evaporate the excess of acid completely before dissolving in water. Without doing this it was impossible to effect a good precipitation of the basic sulphate.

## Manchester Section.

Meeting held at Grand Hotel, December 3, 1920.

MR. JOHN ALLAN IN THE CHAIR.

### THE ORIGIN, DEVELOPMENT, AND VALUE OF THE THALLEIOQUIN REACTION.

BY WM. BEAMONT HART, F.I.C.

(Abstract.)

An investigation as to the analytical value of the thalleioquin reaction was carried out by the author in the year 1909, but the results as to its quantitative value were of such a negative character that they were not considered to be worthy of publication at the time; in view, however, of later published work, they appear to have some importance.

The earliest published record of the green coloration produced by the addition to a quinine solution of chlorine water followed by ammonia is made by

H. A. Meeson (Phil. Mag., 1835, 158), who gave its sensibility as 1 in 8750. In Merck's "Reagentzien Verzeichnis" Manson's reaction for morphine and quinine is mentioned, but from the description and references it is quite evident that the name "Manson" is a misprint for "Meeson." Brandes and Leber, 1839, gave the name of dalliochin or thalleioquin to the green product, hence the name of the reaction.

The reaction has been developed in various ways too numerous to detail here, by substitution of bromine for chlorine or by different methods of liberating halogen, to a sensitiveness of 1:20,000.

Attempts have been made to use this reaction for the colorimetric determination of quinine, using varying quantities of halogen per quinine molecule: Trimble<sup>1</sup> used 38 atoms of chlorine; Léger<sup>2</sup> used 13 atoms of bromine, and states that the method is useless; Vondraseck,<sup>3</sup> using potassium bromate and hydrochloric acid, assumed that with modification and practice it could be used; La Wall used from 10 to 40 atoms of bromine from potassium bromate and hydrobromic acid per mol. of quinine.<sup>4</sup>

From the figures given by Trimble and Léger—the only data found up to 1909—it appeared that the amount of halogen used was excessive and variable. The present author therefore investigated the reaction to determine more minutely (1) the exact conditions necessary, (2) its limit of sensitiveness, and (3) if it were possible to base a quantitative method on it, using bromine as the halogen.

The following solutions were used: (1) Quinine as sulphate, at dilutions 1:1000 to 1:200,000; (2) bromine water, freshly made; and (3) ammonia, 5% solution. Preliminary trials showed that the shade of colour produced depended on (1) the quantity of bromine, (2) time of action of bromine before addition of ammonia, and (3) deterioration of colour by standing after reaction is obtained.

**Quantity of bromine.**—In all, eleven dilutions were treated with increasing amounts of bromine, starting with 0.75 and increasing by 0.75 up to 12.75 atoms per mol. of quinine, ammonia being added after similar times of action, the solutions made up to a total volume of 25 c.c., or 2½ inches in the tube, and the colour shade noted, with the following results:—

With quinine solutions 1:1750 or stronger, and with 6 or more atoms of bromine per mol. of quinine, before ammonia addition, a yellow precipitate is obtained.

With increase of bromine and addition of ammonia the colour proceeds from blue, blue-green to yellow-green and yellow; finally this yellow disappears, except in solutions 1:5000 or stronger.

The strongest colour depth is obtained with 6 atoms of bromine shown consecutively in dilutions 1:1750 to 1:1,200,000; at 1:1000 this maximum appears somewhat earlier with 5.25 atoms of bromine, but the difficulty of comparing colours of precipitates may account for this.

The stronger the quinine solution, the earlier does the yellow shade of green appear; at 1:1000 to 1:10,000 it appears before and extends longer in the series after the maximum colour is attained.

Contrary to a published statement<sup>5</sup> that no green precipitate is obtained in quinine solutions of greater dilution than 1:1000, these results show at 1:5000, with 6 atoms of bromine, a decided cloudiness; at 1:2500, with the same amount of bromine, a precipitate; at 1:1750 this precipitation begins at 0.75 atom, is distinct at 2.25 atoms, and increases up to the addition of 6 atoms of bromine.

During the addition of ammonia, at dilutions of 1:1000 to 1:5000, a pink coloration is first obtained, which appears earlier with increase of



quinine concentration; in every case, when the solution is alkaline, this coloration changes to green on standing.

With increase of bromine, at dilutions of 1:1000 to 1:5000, during addition of ammonia, white fumes appear, which with increase of quinine appear earlier in the series; these fumes were noted with bromine present to the extent of about 0.023 g. per 100 c.c. of solution in excess of the 6 atoms per quinine molecule.

*Time of action of bromine before addition of ammonia.*—Experiments were now made to find the influence of definite intervals of time of action of bromine before addition of ammonia, the intervals chosen being immediate (five seconds), one minute, and five minutes, with the following results: With immediate action, and addition of ammonia till the solution is alkaline, the best results are obtained with stronger solutions, down to 1:40,000, according to the amount of bromine present; with one-minute action weaker solutions give the best results; action of bromine for five minutes reduces the resultant colour strength, but in the weaker solutions (1:40,000 downwards) this is still of deeper shade than with the corresponding dilutions with immediate action.

*Colour-strength deterioration.*—Trials made for this purpose showed that the green colour fades to some extent, but not very materially up to five minutes.

The following conclusions are drawn from the above results: (1) Excess of bromine and its prolonged action are detrimental to the production of the thalleioquin reaction, 6 atoms of this halogen for one minute being ample for strongest colour depth; (2) with quinine solution 1:1000 to 1:20,000 the reaction commences in presence of 0.75, at 1:40,000 to 1:80,000 in presence of 1.5 to 2.25, at 1:120,000 in presence of 3, and at 1:200,000 in presence of 6 atoms of bromine per mol. of quinine; (3) as a qualitative reaction, by making a few preliminary trials it can be made very sensitive, the limit being 1:250,000 in a depth of liquid  $2\frac{1}{2}$  inches (of course, the greater the depth the greater will be its sensibility); for quantitative work, with so many variables, no credence of even approximate accuracy can be assigned to the reaction, except under very strict conditions and in very dilute solutions, as shown.

*Bromine absorption in aqueous solution.*—Attempts were made to determine the bromine absorption of quinine in aqueous solution for 5, 10, and 20 minutes' action with 2.5, 5, and 11.5 atoms of bromine per mol. of quinine, using quinine at 1:1000 and bromine water at one-tenth saturation, the excess of bromine being titrated with thio-sulphate after addition of potassium iodide. The results obtained showed that although four atoms of bromine appears to be the maximum quantity absorbed, and this is constant for five minutes, yet any excess over two atoms is only loosely held and appears to be slowly given up again.

The earlier workers (Andre, Brandes, etc.) did not find chlorine in their product; Commanducci,<sup>1</sup> by varying the time of action of chlorine, obtained two new red products even in alkaline solution—a statement at variance with the present author's bromine results. Comstock and Koenigs,<sup>2</sup> by extracting their quinine dibromide with water and separating by means of ammonia, did not obtain any thalleioquin reaction. Bnraczewski and Dzinr-zynski,<sup>3</sup> on treating their quinine pentabromide with water not above 40° C., filtering and adding ammonia to the filtrate, obtained a white flocculent precipitate which soon became emerald-green and contained bromine, and they state that it is probably identical with the substance formed in the thalleioquin reaction. Christensen,<sup>4</sup> by progressive

chlorination of quinine, obtained (1) quinine hydroxychloride and dichloride, in which the vinyl group is saturated and which give the thalleioquin reaction when chlorine followed by ammonia is added to the slightly acid solution; (2) with 4 atoms of chlorine, an unstable base, 5-chloro-6-hydroxy-cinchonine hydroxychloride, which gives the thalleioquin reaction on treatment with an oxidising agent, a substance that combines with chlorine, and ammonia; (3) with 6 atoms of chlorine, an unstable base, 5-dichloro-6-ketocinchonine hydroxychloride, which liberates two atoms of iodine from potassium iodide for the one active chlorine present and therefore probably contains the group  $-\text{CO}.\text{CCl}_2-$  in the quinoline nucleus, with consequent conversion into the group  $-\text{C}(\text{OH})\text{CCl}_2-$ . This base gives the thalleioquin reaction on addition of ammonia only; the green product on drying leaves a residue of chloro-5,6-diketohydroxy-cinchonine, the group  $-\text{CO}.\text{CCl}_2-$  being replaced, in presence of ammonia, by the  $-\text{CO}.\text{CO}-$  group.

As far as is known, then, the action of chlorine or bromine on quinine in dilute aqueous acidified solution is similar, and the products, beyond the di-halogen stage, that can be formed during or can give rise to the thalleioquin reaction, are numerous. The author attacked the problem from the analytical standpoint, and his conclusion, in 1909, that 6 atoms of halogen are required for the full reaction, is confirmed by the later results of Christensen; these also explain why, in the results of the bromine absorption in aqueous solution, apparently not more than four atoms as a maximum are absorbed, whilst actually six atoms take part in the reaction, the two atoms of iodine set free being determined as unabsorbed bromine. It follows, then, that quinine may be determined from its bromine absorption for five minutes in the manner stated: 1 c.c. N/10 Br = 0.0081 g. quinine.

It is possible and probable that the progressive halogen action runs concurrently to some extent; that is to say, part of the halogen is preferably used in further conversion of the intermediate products than in full conversion of quinine into intermediate products; this would explain why the thalleioquin reaction is obtained, though not to the full extent, by the action of much less than six atoms of bromine per mol. of quinine. Christensen's statement that the intermediate product, 5-chloro-6-hydroxy-cinchonine hydroxychloride, gives the reaction by treatment with an oxidising agent, a substance that combines with chlorine, and ammonia, may explain the mechanism of the delay in the final green colour production by Vogel's reaction (1850) and also by Battandier's reaction (1904), since we have the three essentials, time only being necessary; to some extent this also applies to Kletzinsky's reaction (1854), though in this case a further change probably occurs. During the early stage of this investigation the use of a solvent, such as amyl alcohol, for the green product suggested itself, but from the above results it was not thought to be of any value.

- <sup>1</sup> Flückiger, 1872; Vulpius, Vitall, 1886; Hyde, 1897, etc.
- <sup>2</sup> Allen, "Conm. Org. Anal." 2nd ed.; vol. 3, pt. 2, pp. 401-402.
- <sup>3</sup> Léger, J. Pharm. Chim., 1904, 281.
- <sup>4</sup> Morck, Ann. Rep., 1908, 275.
- <sup>5</sup> La Wall, Amer. J. Pharm., 1912, 454.
- <sup>6</sup> Commanducci, Chem. Zentr., 1910, 1885.
- <sup>7</sup> Ber., 1892, 1539.
- <sup>8</sup> Ann. Akad. Wiss. Krakau, 1909, 333.
- <sup>9</sup> Ber. Douts. Pharm. Ges., 1915, 250; 1916, 247.

#### DISCUSSION.

The AUTHOR, replying to a question, said that it was not practicable to estimate the colour depths by the absorption spectra or by the colorimeter, owing to the rapid changes of colour of the solutions and the large number of experiments being carried out at the same time.

## Yorkshire Section.

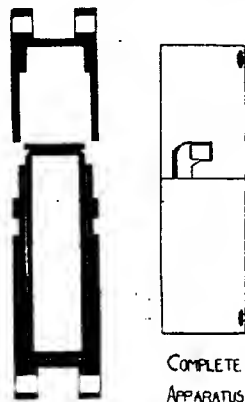
Meeting held at the Queen's Hotel, Jan. 17, 1921.

DR. L. L. LLOYD IN THE CHAIR.

### NOTE ON AN APPARATUS FOR SODIUM PEROXIDE FUSIONS.

BY H. J. HODSMAN, M.B.E., M.Sc., F.I.C.

The method of fusion with sodium peroxide for converting insoluble combustible matter into a form suitable for ordinary analytical operations is finding many applications, viz., in the determination of sulphur, chlorine, and phosphorus. The forms of apparatus described hitherto have not been free from objection and even risk in handling, especially to the novice, for the reaction is mildly explosive. In some methods the risk is increased by immersing the apparatus more or less completely in water. Screwed threads are liable to seize, making the apparatus difficult to open. The trouble is accentuated if any of the melt solidifies in the threads. Firing by means of heated wires or pellets is erratic, especially to the inexperienced.



The apparatus described below has been designed to avoid these shortcomings. The reaction vessel consists of a thin-walled cartridge of steel tube with a well-fitting cap, having so small a surface of contact that a joint cemented by solidified melt can readily be broken or freed by solution in water. This cartridge is enclosed in an outer cylinder, the lid of which is held in place by a bayonet catch which, when drawn up, holds the cap of the inner tube in place. The cartridge is charged and placed in the outer case, the lid is fitted on, and the apparatus is supported inclined in a clamp and heated at the base for a few minutes over a Bunsen flame until firing has occurred. Alternatively, the apparatus may be placed in a muffle. After cooling, the lid is removed, the cartridge withdrawn, its contents are extracted with water, and the analysis can be proceeded with by the methods in vogue for the determination in question. The outer tube and cover are extended at both ends and bored for the insertion of a rod which facilitates opening. This is unnecessary when the apparatus is properly used. The apparatus can be opened while hot if desired.

Fuel Department, Leeds University.

#### DISCUSSION.

Mr. W. McD. Mackey said that when using sodium peroxide in a Roland Wild calorimeter very

high results had been obtained in a number of cases, which he attributed to the possible presence of free sodium in the peroxide.

Mr. D. McCannlish asked if the peroxide fusion method could be used for the determination of arsenic in fuels, and also whether the author prescribed any special degree of fineness for the powdered fuel.

The Author, in reply, said that he had not come across any sodium peroxide which contained free sodium, and he suggested that Mr. Mackey's high results might have been due to leakage of water into the crucible. The peroxide fusion method was especially useful for the estimation of sulphur in coal, but there was no apparent objection to using it for arsenic determinations. He had used it for the estimation of phosphorus in coke. As regards the fineness of the coal powder no special precautions were needed if the powder and peroxide were well mixed. The fact that the whole apparatus was directly heated in a Bunsen burner made it unnecessary to have such an intimate and homogeneous mixture as was required in a sodium peroxide calorimeter. In the author's experience the peroxide calorimeters of Parr gave fairly satisfactory results for the ordinary grades of coal.

## Communication.

### THE HYDROLYTIC ALKALINITY OF PURE AND COMMERCIAL SOAPS.

(See J., Feb. 28, 1921, 27—29 t.)

Mr. R. G. SARAIYA writes as follows:—On p. 29 t, under "Sodium carbonate," it is said:—"Simmons states that 'sodium carbonate has an irritating effect . . . and must only be present to a very small extent in a toilet soap, usually not exceeding 0.15%.' The experiment gave a concentration of hydroxyl ion in 0.15% sodium carbonate solution at 25°, amounting to 0.0010 N. . . . Thus a maximum alkalinity recognised as permissible is about 0.001—0.002N OH' . . . ' Now, Simmons refers to 0.15% of sodium carbonate on the soap. The determination of the hydroxyl concentration in the above experiment was made on a 0.15% solution of sodium carbonate. Hence it seems scarcely justifiable to conclude from this experiment that 'a maximum alkalinity recognised as permissible is 0.001—0.002N OH'." It would be interesting in this connexion to find the OH' concentration in aqueous solution of a pure soap containing 0.15% (on the soap) of sodium carbonate."

Mr. T. R. BOLAM writes in reply:—"We are indebted to Mr. Saraiya for having pointed out the obvious slip in the short final paragraph dealing with sodium carbonate, which fortunately leaves the experimental data and the paper itself otherwise unaffected. 0.15% sodium carbonate in a soap in the usual technical units amounts to about 1.6% of excess of alkali in the form of carbonate when expressed in chemical equivalents. The data show that sodium carbonate taken alone is much more hydrolysed than an equivalent quantity of soap. Previous data show that in N/20 or N/10 sodium palmitate solutions the alkalinity is 0.0011 to 0.0014 N OH', and that when to these solutions there is added 1 or 2% (in equivalents) of sodium hydroxide the concentration of hydroxyl ion is increased by about half the normality of the excess of alkali added. This would indicate that addition of even these small quantities of sodium carbonate would increase the hydroxyl concentration of 1% soap solutions by an appreciable fraction."

## Edinburgh Section.

Meeting held at Edinburgh on Feb. 15, 1921.

DR. D. S. JERRAN IN THE CHAIR.

## INVESTIGATIONS ON THE RANCIDITY OF BUTTER AND MARGARINE FATS.

BY W. N. STOKOE, B.Sc., A.I.C.

Of all the problems which those concerned with the manufacture of fatty foods have to face the question of the occurrence of rancidity, either in the raw materials or the finished article, is probably the most acute. During the warm summer months in particular the manufacturer is constantly haunted by the possibility of his goods going wrong within a short time, in spite of meticulous care during manufacture, and each year an astonishingly large quantity of unsaleable goods finds its way eventually to the soapmaker. It is, therefore, of paramount importance that this problem should be carefully studied, and a thorough insight gained into the conditions causing and favouring the occurrence of rancidity.

A considerable amount of work has already been done and published on this subject, but even yet the nature of the chemical changes covered by the term rancidity is but little understood, and on many points there is still considerable diversity of opinion.

The rancidity of pure fats is now generally understood to be due to a product of hydrolysis and oxidation, and it is usually characterised by the loss of the natural sweetness and flavour of the fat, the formation of compounds of unpleasant odour and taste and a development of acidity in the fat. However, the acidity of a fat is in no way a measure of its rancidity or non-rancidity, for it is possible to have examples of fats showing extremely low acidity and yet being very rancid, whereas other cases may occur in which the fats show a much higher free fatty acid content and yet are perfectly sweet and non-rancid.

In commercial oils and fats the chief causes influencing rancidity seem to be the action of moisture, air, and light, assisted by traces of lipolytic enzymes which may be present in the oils. Generally speaking vegetable oils and fats are free from these enzymes owing to the comparatively high temperature to which the oil is subjected during refining. It is a fact, though, that under certain conditions lipolytic enzymes can withstand a considerably higher temperature than the usual thermal destruction point of enzymes without being destroyed.

Animal fats, on the other hand, being rendered at a low temperature, contain traces of enzymes derived from the tissues of the animal. The presence of enzymes will largely depend on the care taken to ensure separation of tissue.

The first stage in the development of rancidity is hydrolysis, which, however, may be very slight and scarcely capable of detection by chemical means; then oxidation and a multitude of other reactions take place. Some observers state that the oxidation of the glycerol set free in the initial hydrolysis is the actual cause of the rancid taste.

It has been thought that micro-organisms are the cause of rancidity, but in pure fats this is not the case, for organisms introduced into the fat quickly die. In butter and margarine the case is different, for here there is adequate nidus for the development of the organism. Ritsert\* found that pure lard was

not turned rancid by bacteria, aerobic or anaerobic, and this result has been confirmed by other workers. Duclaux\* showed that micro-organisms play no part in the production of rancidity, for the fats being insoluble in water cannot afford nutriment to the cells. The present author's experiments have confirmed this view, though it would appear that a slight hydrolysis does occur in the case of the fat-splitting organisms.

A culture was taken from a sample of rancid margarine showing a free fatty acid content of 1.84%, and the principal organisms present plated out and obtained in the pure culture state. Samples of pure fats were procured and inoculated with the pure cultures. In these experiments the organisms used were a mould, *Penicillium glaucum*, and a bacillus, *B. fluor. non-lig.* After a week the free fatty acid was determined in a portion of the inoculated fats, the remainder being replaced in the incubator. After a second week the acidity was again determined.

TABLE 1.  
Inoculated with *Penicillium glaucum*.

Oil or fat.	Free fatty acid.	After 1 week.	After 2 weeks.
(a) Coconut .. ..	0.078	0.085	0.085
(b) Palm kernel .. ..	0.078	0.085	0.085
(c) Lard .. ..	0.20	0.22	0.23
(d) Oleo .. ..	0.25	0.26	0.26
(e) Prem. ius .. ..	0.16	0.19	0.19
(f) Groundnut .. ..	0.28	0.28	0.29
(g) Soya bean .. ..	0.19	0.19	0.19
Control 1—Coconut ..	0.078	0.078	0.078
Control 2—Oleo .. ..	0.24	0.24	0.26

Note.—The free fatty acid is expressed in terms of oleic acid. In all cases the odour and taste were unimpaired.

Table 1 shows the free fatty acid determinations on the fats inoculated with *Penicillium* and two uninoculated controls. The figures for the fats inoculated with *B. fluor. non-lig.* are similar. The results show a slight increase in acidity, whereas there is no appreciable increase in the control samples. As far as taste and odour were concerned the fats had apparently not deteriorated in any way. The fat remaining after the second series of determinations was emulsified with nutrient media and placed in the incubator. In each case after a few days a growth of the mould organism was obtained, showing that the spores of the mould remained fertile in the oil. This point will be referred to again later.

It appears probable that the vegetative cells live for a short time in the fat while the organism uses up its reserve material. In this period the organism presumably secretes a small quantity of lipase which is able to cause a slight hydrolysis of the fat. It is, of course, well known that with enzyme-secreting organisms the maximum amount of enzyme present in the plant is obtained when the plant begins to utilise its reserve material. It is then that the enzyme is best able to diffuse from the cells of the plant.

The rancidity of butter and margarine is almost wholly caused by the action of micro-organisms, which find in these substances a suitable medium for their growth and propagation. To a certain extent the development is restrained by the use of preservatives such as salt and boron compounds, but at best these are but feeble antiseptics in the quantity usually found or allowed in foodstuffs.

The kinds or forms of rancidity which occur are several and may be classified into divisions depending largely on macroscopic appearance, odour, and taste.

\* Inang, Dissert., Berlin, 1890.

\* Ann. Inst. Pasteur, 1887; Comptes rend., 102, 1077.

(1) *The fat having a stale greasy taste and odour, but not discoloured or visibly affected in any way.* Instances of this type of rancidity are found principally with pastry margarines, i.e., margarines of a hard nature containing mostly fats of high m.p. These are, as often as not, churned with water only, not milk. This case is comparatively simple, and is really analogous to the rancidity of a pure fat, where the rancidity changes are due to air, light, and moisture, possibly assisted or accelerated by the action of traces of lipolytic enzymes present in the fats. The author has attempted to determine whether or not fats rancidify more quickly than the same fats previously heated to a temperature sufficient to destroy any enzyme present. These experiments were, however, unsatisfactory, and although a good many figures were obtained it would be unwise to attempt to draw any conclusions from them. Generally speaking heated oils increased in free fatty acid content more rapidly than the unheated oils, so that enzyme action is altogether out of the question as far as these experiments go. Of course pastry margarines which contain, as a rule, a major proportion of premier jus or stearine, require to be churned at a high temperature approaching the thermal destruction point of enzymes, and it would therefore be expected that any enzyme present would be considerably weakened, if not totally destroyed. In general the flavour of animal oils and fats is impaired by heat.

Samples of margarine rancid in the form just referred to were invariably old and in most cases had been unduly exposed to air and warmth. The fat had a stale taste and tallowy odour, but did not show an abnormal acidity on analysis. The outside of the margarine was much more affected than the inside.

The "tallowiness" of old butter is another instance of this form of rancidity, seeing that it is not caused by biological conditions, but by the action of air and light.

It seems that this characteristic tallowiness of rancid fats and rancid butter is an oxidation effect. Hunziker and Hosman\* suggest that glycolic acid is formed from the glycerol produced by the initial hydrolysis and that the glycolic acid then combines with fatty acids, so producing the unpleasant flavoured compounds. The presence of lactose intensifies the oxidation effect since it leads to excess of glycolic acid. Light does not appear to be essential to the oxidation but has an accelerating effect. Oxidation is accompanied by a decrease in the iodine value.

(2) *The "perfume" form of rancidity.* For want of a better description this form of rancidity, occurring frequently in margarine, may be termed the "perfume" rancidity. A strong distinctive aromatic odour is produced which is not at all offensive, but the rancid fat has a vile pungent taste.

This form of rancidity is most obnoxious, for it can occur in margarines only a few days old. The margarine may appear perfectly good and wholesome and yet have this most objectionable taste and powerful odour. In more advanced stages of this rancidity the margarine is often discoloured and patchy, but it is usually the early stages with which one has to deal.

In the investigation of this problem a number of rancid samples were procured and biological cultures prepared from each, after which the principal organisms present were isolated in the pure culture state. At this point one fact became evident, namely, that in every case mould organisms, principally *Penicillium* species, were present. Another noticeable feature is the fewness of the lactic acid organisms present on the plates.

Analysis of the fat of the samples showed that in every case in which this type of rancidity was observed the fat contained a proportion of coconut oil, palm kernel oil, or both. This directly confirmed manufacturing experience, which always associated coconut oil with and blamed it for this trouble.

It was next decided to prepare media containing pure oils and to inoculate tubes of the special media with the organisms isolated in the former work. The media were prepared by emulsifying nutrient lactose, gelatin, and agar with sterilised fats, and in this way media were made up containing respectively coconut, palm kernel, and other common fats. Tubes of these media were inoculated with the different organisms previously obtained and incubated at 68° F. Table 2 shows the results obtained.

TABLE 2.  
Incubation: 7 days at 68° F.

Organism.	Wild yeast.	<i>B. acidilactici</i> .	<i>Sarcina lutea</i> .	<i>Penicillium glaucum</i> .
Coconut oil media	No odour	No odour	Putrid odour. Decomposition of nitrogenous matter.	Very strong "perfume" odour.
Palm kernel oil media	No odour	No odour	Do.	Strong "perfume" odour, same as C.N. Musty odour.
Media containing oleo and lard.	Slightly rancid odour.	No odour	Do.	
Media containing cotton seed oil.	Very slight odour suggest stale coo-	No odour	Do.	Slight musty odour.
Media containing arachis oil.	Do.	No odour	Do.	Do.
Media containing soya bean oil.	Do.	No odour	Do.	Do.

It will be observed that only the coconut and palm kernel media produced the "perfume" in question, and that the organism concerned was *Penicillium*. Later on a sample of babassu fat was examined in the same way, and was found to give a similar result to coconut and palm kernel. These results were confirmed by repetition using samples of oil of various origin. In every case the mould *Penicillium glaucum* was able to produce this "perfume" rancidity with coconut and palm kernel oils. Up to the present *Penicillium glaucum* was the only mould organism experimented with, seeing it was the principal organism found in the samples examined. Subsequently, however, other mould organisms were isolated from rancid butter and margarine. Pure cultures of these were prepared in the usual way.

Table 3 shows the effect of cultivating the various common mould organisms on media containing coconut oil and butter fat.

It appears from this table that species of *Penicillium* and *Aspergillus* alone can produce the "perfume" odour, but that hydrolysis occurs to a greater or less degree in each case.

The "perfume" odour is not produced with butter fat.

It will be noticed that *Oidium lactis* liquefied the media. This, of course, only occurs in the case of acid media.

In a recent paper on this same subject, H. C. Jacobsen,\* who has worked along similar lines, states that the ester odour of rancid coconut oil appears only under microbic influence. He finds

\* J. Dairy Sci., 1917, 1, 320.

\* Follen Microbiologen, Deel V., Aft. 2 (Nov., 1918).

further that only the following moulds have this capacity:—*Penicillium glaucum*, *Aspergillus* spec., *Mucor* species, *Cladosporeum* species, *Hormodendron* spec., *Phoma* spec., and *Cladosporeum butyri*. *Oidium lactis* does not cause rancidity.

TABLE 3.

Incubation: 7 days at 68° F. Coconut oil (a), f.f.a. 0.07% lauric. Butter fat (b), f.f.a. 0.33% oleic.

Organism.	Coconut oil media.	(a)	Butter fat media.	(b)
	Observations.	F.f.a.	Observations.	F.f.a.
<i>Oidium lactis</i>	Cheesy, not unpleasant odour, gelatin liquefied.	13.6	Strong cheesy odour.	10.3
<i>Penicillium glaucum</i>	Strong "perfume" odour, gelatin liquefied, fat discoloured.	6.1	Slight odour of butyric acid, also somewhat mouldy odour.	3.1
<i>Aspergillus glaucum</i>	Strong "perfume" odour, gelatin liquefied.	6.8	Rancid odour, somewhat masked by smell of mould.	5.2
<i>Botrytis cinerea</i>	Musty earthy odour, gelatin not liquefied.	4.7	Odour very slight, not distinctive.	4.8
<i>Cladosporeum oidium</i>	No appreciable odour, gelatin not liquefied, fat discoloured; black.	2.0	Slightly stale odour, fat much discoloured.	6.5
<i>Mucor mucedo</i>	Earthy odour, gelatin not liquefied.	9.6	Stale odour of rancid butter.	6.8

Fatty acid expressed as (a) lauric acid, (b) oleic acid.

Jacobsen does not state the precise organism used in his experiments. Certainly *Botrytis cinerea*, *Cladosporeum oidium*, and *Mucor mucedo* did not produce the "perfume" form of rancidity.

Experiments were next undertaken to attempt to determine the conditions favouring the production of this "perfume" rancidity.

#### Influence of the composition of the media.

The following media were prepared, sterilised and emulsified with various oils and fats as before. (a) Nutrient broth—peptone, flesh, lactose. (b) do., no sugar. (c) Nutrient gelatin, no flesh, dextrose. (d) Milk. (e) Whey soured by pure lactic acid ferment. (f) Casein. Portions of these media were inoculated with the pure cultures previously prepared and incubated for a week at 68° F. (20° C.).

Tables showing the individual observations were not drawn up, but briefly the principal points observed were:—

(1) In every instance the growth of species of *Penicillium* or *Aspergillus* upon media containing coconut or palm kernel oils produced the "perfume" form of rancidity.

(2) Of the two oils coconut seems to be the more susceptible; throughout the whole series of observations, as far as could be ascertained, the odour from the coconut media was stronger than from the palm kernel, though identical in character.

(3) The influence of sugar in the media is noteworthy; in absence of sugar the odoriferous substance is not so readily produced.

(4) Mould spores did not readily germinate in the soured whey media, probably owing in some measure to its nitrogen deficiency, but with the casein media there was a prolific mould growth and corresponding rancidity in the course of a few days.

Further experiments were next undertaken to ascertain the effect of acidity or alkalinity of the media. Table 4 shows the relative quantities used and the results of the experiments.

TABLE 4.

Organism: *Penicillium glaucum*. Media: Coconut oil (f.f.a. 0.07%), gelatin lactose.

Acidity or alkalinity of media.	Observations after 7 days incubation at 68° F. (20° C.).	F.f.a. as lauric acid.
21% N/10 HCl.	Abundant mould growth; media liquefied, "perfume" odour; fat discoloured green.	4.9
5% N/10 HCl.	Less mould growth; media not liquefied; "perfume" odour; discoloration very slight.	2.0
5% N/1 HCl.	Much growth of the mould; odour very slight; fat orange coloured.	4.2
5% N/10 NaOH.	Abundant growth; strong "perfume" odour; gelatin liquefied.	5.4
5% N/1 NaOH.	Less luxuriant growth; gelatin liquefied; odour very slight.	3.2

The experiments are not very conclusive, but it would appear that the "perfume" is produced even in presence of 5% of N/1 acid. In presence of a similar quantity of alkali the perfume is produced to a small extent, but in both cases a considerable hydrolysis of the fat takes place.

*The influence of temperature.*—This is, of course, very marked. Warmth accelerates the growth of the organism, and for this reason it is really only in summer that the problem of the occurrence of this rancidity is accentuated. In winter the preservatives present in margarine are usually sufficient to restrain undue propagation of the organisms present.

Samples of margarine showing this "perfume" form of rancidity are invariably more rancid at the outside than the inside. As often as not the inside is perfectly sweet and fresh. In more advanced stages the rancidity does penetrate right down through the bulk, but invariably it will be found that the outside is the more decomposed. The free fatty acid content is always found to be much higher at the outside. The "perfume" rancidity is always accompanied by fat splitting to a greater or less degree. This is, of course, to be expected seeing that both *Penicillium* and *Asper-*



This layer of rancid fat showing disintegrated mycelium of the mould organism. ( $\times 50$ , unstained.)

FIG. 1.

*gillus* are lipase-secreting organisms. Microscopical examination of the rancid margarine often reveals the presence of mycelia which penetrate through the fat (see Fig. 1).

The mycelia are often much disintegrated and in samples but slightly rancid their presence is sometimes extremely difficult to detect. The amount of the compounds produced giving the strong perfume is exceedingly small. Steam distillation of 200 g. of a rancid margarine only yielded less than 0.1 c.c. of the odoriferous substances.

The question as to the mechanism of the action of this form of rancidity now arises. Apparently air assists the action if not necessary to it. Does lipase so act on coconut and palm kernel oils to produce the effect in question; or is the lipase of the *Penicillium* and *Aspergillus* organisms peculiar to itself; or is another independent enzyme responsible for the acceleration of the change? When coconut or palm kernel oil rancidifies through the usual agency of air, light and moisture the "perfume" is not produced; there is produced, on the other hand, the usual stale greasy taste and odour common to rancid fats. Clearly then an additional factor enters into consideration.

In the first instance a solution containing lipase of vegetable origin was prepared from castor oil seeds. Germinating seeds were ground up in a mortar with a 5% sodium chloride solution containing 0.2% potassium cyanide, the whole being allowed to stand after the operation for 24 hours. The filtered solution was then ready for the experiments. Portions of coconut and palm kernel oil were mixed with the enzyme solution, four drops of carbon tetrachloride added, and the test mixtures kept for given periods of time at a temperature of 68° F. In a series of experiments the time, concentration and proportion of enzyme liquid were varied. In all cases hydrolysis of the fat took place to a greater or less extent, sometimes considerable, but there was no production of aroma or "perfume" whatsoever, showing that the lipase alone as prepared from castor seed is incapable of producing the effect in question.

Attempts were next made to prepare the enzyme or enzymes of the mould *Penicillium glaucum*. Several methods were tried without success, but the following method proved fairly satisfactory. A wide vessel was filled to a depth of about  $\frac{1}{2}$  in. with a medium made by emulsifying coconut oil with a gelatin lactose medium. This was liberally sown with spores of the organism and placed in the incubator. After several days a luxuriant growth of the mould completely covered the surface of the medium. The latter was then gently warmed to melt it completely and enable the mould plant to be lifted bodily out of the liquid. It was then transferred to a vessel containing distilled water, which was replaced in the incubator. A small amount of the original medium adhered to the plant and was more or less dissolved by the water, forming a substratum for the organism of very low nutritive value. After several days the plant was removed from its watery bed and the liquid filtered several times through cotton wool until free from cellular elements as determined by microscopical examination and incubation tests. Addition of strong alcohol to the solution produced a slight opalescence. Tested with tincture of guaiacum and hydrogen peroxide, a positive reaction was obtained, but the boiled solution failed to give the reaction.

The liquid containing the enzymes was next mixed with sterilised coconut oil in varying quantities with the addition of four drops of carbon tetrachloride, and the tests set aside in the incubator at a temperature of 68° F. A control was also prepared, in which the enzyme liquid had previously been boiled. After a week it was found that the "perfume" odour had been produced and that the fat tasted very rancid, but the control was still sweet to taste, and there was no suggestion of the "perfume" odour. A similar series of tests with the further addition of lactose was made, the results of which confirmed the former. If anything

the "perfume" odour was stronger in the last experiments. A similar test with the addition of lactose was made with the castor oil seed lipase solution, but there was no formation of the "perfume" rancidity. Table 5 shows the results obtained, using 10 g. of coconut oil in each case.

TABLE 5.

Lipase soln. from castor oil seeds.	Lipase soln. from <i>Penicillium glaucum</i> .	Lactose.	Observation.	Free fatty acid as lauric acid.
10 c.c.	—	Nil	No formation of	0.89
20 c.c.	—	Present	odoriferous substances; the fat tastes slightly stale and "off."	1.53
30 c.c.	—	Nil		1.84
100 c.c.	—	Nil		5.68
20 c.c. Boiled.	—	Nil	No odour, taste still sweet and fresh.	0.12
—	10 c.c.	Nil	"Perfume"	1.29
—	20 c.c.	Nil	odour produced.	2.00
—	10 c.c.	Present	strong rancid taste.	1.12
—	20 c.c.	Present		2.38
—	20 c.c. Boiled.	Present	No odour; taste still quite sweet and fresh.	0.16

It will be observed that the extent of the hydrolysis brought about by the enzyme is roughly proportional to the quantity of enzyme liquid used.

These results appear to indicate that the "perfume" form of rancidity of the coconut group of oils is brought about by an enzyme secreted by certain of the mould organisms acting probably in conjunction with the lipase secreted by the same organism. Up to the present I have not succeeded in preparing this enzyme apart from lipase, nor yet obtained much reliable information concerning its action. It seems, however, to provide a subject for an interesting piece of biological research. Jacobsen in his paper, already referred to, calls the "perfume" produced an "ester odour," and appears to regard it as due to esters of some of the volatile acids. In this he seems to have been mistaken, for although there may be traces of esters present in the substances produced, undoubtedly the greater part is a ketone or mixture of ketones. About 75% is methyl nonyl ketone, and this is accompanied by methyl heptyl and methyl undecyl ketones. By steam distilling a culture of *Penicillium* grown on a medium containing coconut oil a small quantity of these odoriferous substances was obtained in a comparatively pure state. The distillate gave a strongly positive reaction for ketones, but was negative to tests for aldehydes.

### 3. Rancidity where there is a marked discoloration of the butter or margarine.

As already indicated, fats in the advanced stages of perfume rancidity often show discoloration. The yellow colour of the fat loses its natural brightness and sometimes the fat tends to lose its colour. Sometimes darker coloured patches are formed. Some time ago dimethylaminoazobenzene was largely used to colour margarine, but of late it has been more or less superseded owing to the action of rancidifying fat upon it. The dye invariably tended to change its shade to a pinkish hue as the formation of free acidity proceeded.

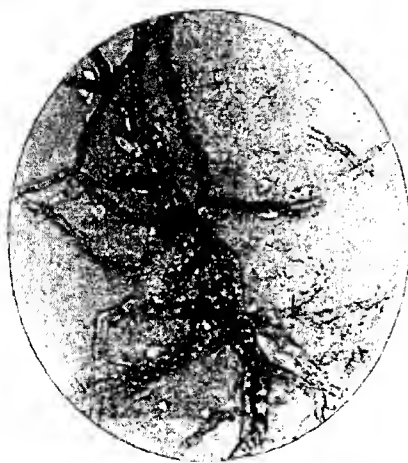
The discoloration of the rancid margarines may take the form of black irregular patches, blue patches, or large spreading pink to red areas dotted over the bulk of the margarine (this must not be confused with the dye effect already mentioned). Sometimes one form predominates, sometimes another, or they may occur together in the same sample.

All these effects are caused by the growth of micro-organisms. In some cases the organisms are



moulds, in others, bacteria or yeasts. By making cultures from the rancid samples showing these effects the responsible organisms were isolated and studied for identification.

The black patches as a rule do not extend to any depth; they are merely surface colonies of the organism. In such cases the rancidity is localised, only the surface layer being affected. The appearance of the fat is repulsive, but the taste is only slightly affected and no odour is produced. The organism causing this trouble is a species of *Cladosporium*, and by smearing the surface of butter or margarine with a culture of the organism the discoloration could be closely imitated. Examination of discoloured portions of the fat under the microscope shows the mycelium of the organism (see Fig. 2).



Discoloured (black) portion of margarine from surface showing mycelium of *Cladosporium*. (X 400, unstained.)

FIG. 2.

Blue-green patches are caused by *Penicillium glaucum* or like species. Here the rancidity is not so localised for the mycelium of the plant seems to be able to penetrate right down into the fat. Considerable hydrolysis takes place and the effect on the coconut group of fats has already been discussed. In absence of these fats usually a mouldy taste and odour predominate. Examination of the fat shows the mycelium of the organism—often very disintegrated. Owing to the disintegration it is usually impossible to identify the organism at this stage. In many cases it is sufficient to place a very small particle of the fat on a microscope slide and press out flat with a cover slip and then examine with a  $\frac{1}{4}$  in. objective. In less advanced stages of the rancidity it may be necessary, however, to use a larger quantity of the fatty matter and to dissolve away the fat with a solvent. The residue can then be mounted, stained if desired, and examined.

*Aspergillus* species and other common moulds and fungi produce grey to blue-black discolorations, but these organisms do not occur so frequently in butter or margarine. *Penicillium* species are by far the most common.

The red discoloration is caused by one or two organisms obviously of the chromogenic class as well as by certain wild yeasts. At least three different organisms of this nature, *B. prodigiosus*, *S. rosea*, and *M. roseus*, were isolated and identified in the course of this investigation. These are able to cause hydrolysis of the fat to a considerable degree, but no specially marked deterioration in flavour occurs. There is no odour produced.

There are quite a number of other organisms to be found in rancid butter or margarine. Many of these, if not all, are able to hydrolyse the fat to a greater or less degree. In some cases the deterioration in flavour of the fat is marked, in others the taste is but slightly affected. The following organisms were isolated and identified in the course of the work:—*B. fluorescens* liq. and non-liq., *B. proteus* species, *B. coli*, *B. putrificus*, *Bact. punctatum*, *M. luteus*, *S. lutea*, wild yeasts, and torulas, besides a considerable number of species of micrococci and sarcina which were not positively identified. *Sarcina urea* was found on several occasions. In general the organisms belong to the saprophyte class of bacteria.

In the rancidity changes set up in butter and margarine by these organisms, not only is the action on the fat to be considered; there is further the action on the protein matter of the butter or margarine which may be converted into products conferring an obnoxious flavour and aroma on the sample.

It is now well known that the lactic acid organisms themselves have an effect on the keeping properties of butter. Butter prepared from pasteurised cream keeps better than butter prepared from soured cream, other conditions being equal.

In general it is at present impossible to dogmatise as to the definite chemical changes produced by the organisms either in respect to fat or to protein matter; also it is impossible to catalogue rancidity-producing organisms, and in many cases, owing to insufficient description, it is very difficult to identify organisms found.

Just recently a sample of rancid margarine, purple in colour, was examined, from which moulds of the *Penicillium* species were isolated as well as a large fission fungus so far unidentified.

In butter yeast organisms are often found. The action of these is very interesting and possibly capable of being developed to a useful end. A small amount of fat splitting proceeds, and a medium containing coconut oil increased in acidity from 0.1% to 0.8% in a month when inoculated with the organism. A slight, rather pleasant fruity odour is produced alike in most neutral fats. It seems also that certain acid-resisting yeasts have the property of associating themselves with the lactic acid organisms, whereby the life of the latter is prolonged. This appears to have considerable bearing on the flavour-keeping properties of butter and margarine, and samples prepared using mixed cultures under appropriate conditions remained sweet, fresh, and of good flavour for several months, although kept under usually unfavourable conditions.

These investigations have demonstrated the vital necessity of preventing the contamination and invasion of butter and margarine by undesirable rancidifying organisms. A thorough examination into the possible sources of contamination during manufacture was carried out, and in the course of this work some very interesting facts were revealed.

1. *Contamination from air, utensils, machinery, etc.*—This is very important, but it can be reduced to a negligible quantity by thorough cleanliness in manufacturing processes and by sterilising and disinfecting the air in the factory, all utensils, etc.

2. *Raw materials.*—This is perhaps the most serious question of all. Hitherto raw materials have been judged as regards quality on taste, odour, and chemical analysis. As a result of an exhaustive bacteriological investigation it was found that the usual tests are of little value if the oils are not sterile to begin with, owing to the germination of the spores once the material is manufactured and mixed with nitrogenous matter. In addition to the usual tests, therefore, a bacterio-

logical examination of the raw materials used in margarine manufacture is strongly advocated. During the past two years several hundred samples of commercial oils and fats have been submitted to a bacteriological examination. Of these less than 5% proved to be sterile, some 10–20% contained not more than 6 spores per c.c., but the majority simply teemed with spores of mould, mucor, and other spore-forming organisms. Invariably liquid oils were worst in this respect, and so far not a single perfectly sterile sample has been found. Hardened fats proved to be sterile, or practically so; coconut and palm kernel oils were usually fairly sterile, that is to say contained less than 10 spores per c.c., but nevertheless some very bad samples of these oils were encountered.

Animal fats are fairly sterile, but many samples were found to contain bacteria spores rather than mould spores. The vegetable oils usually contain mould, mucor, and fungi rather than bacteria spores.

The following organisms were found to occur most frequently in the oils and fats:—*Penicillium* species, *Aspergillus* species, *Cladosporium* species, *Botrytis cinerea*, *Mucor* species, and a few common sporulating bacteria.

The most satisfactory method of making a bacteriological test on oils and fats was found to be as follows:—Portions of 1 c.c. and 2 c.c. of the oil or just melted fat are withdrawn from the sample by means of sterilised pipettes and run into tubes of nutrient sugar gelatin, the latter being melted. The mouth of the tube having been "flamed," a sterilised stopper is quickly inserted and the mixture of oil and medium violently shaken to produce an emulsion. This is finally poured out into a cold Petri dish, when it will be found that the emulsion sets before any separation of oil or fat occurs. The Petri dishes are then placed in the incubator at a suitable temperature and examined after two or three days, when a count of the colonies present may be taken.

The question of the origin of the mould spores in the oils and fats naturally arises. It would be expected, naturally, that any spores pre-existing in the crude oil would be destroyed during the deodorising process in which high temperatures are employed. It was thought desirable actually to investigate and check this point, seeing that autoclave conditions do not obtain during deodorising, but rather a vacuum is employed. In carrying out this work the author was indebted to several well-known firms of oil refiners who allowed him to visit their works and draw samples at various stages of the refining and deodorising processes, and in particular to one firm who took an active part in the work. The samples so drawn were tested as described. The investigation yielded some remarkable results, which, briefly, were as follows:—

(a) Mould spores existing in the oil prior to deodorising are not invariably destroyed during the process even at a temperature as high as 150°–170° C., the temperature being maintained for several hours. Many spores are destroyed, however, the count being invariably much less after deodorising than before.

(b) Mould spores can remain in a fertile condition in oils for a very long time, only awaiting favourable conditions for development and propagation.

(c) Where filtration is carried out after deodorising, the oil is liable to infection from air and from the filter cloths.

(d) The quality of the seeds or nuts or crude oil has a good deal to do with the relative sterility of the finished oil. With carefully picked seeds or nuts the oil, even before deodorising, is very free from

spores so that the deodorised oil has a better chance of being sterile, or approximately so, than in the too frequent case where the seeds or crude oil are of inferior quality. Undoubtedly the quality of the seeds and nuts largely governs the number of spores in the finished oil.

(e) The use of wooden barrels or containers is much to be deprecated. It is practically impossible to sterilise the wood, and there is always danger of mould development on the wood if it should become damp and have access to air. Further, the canvas which is put on the hungs of the barrels teems with mould spores (see Fig. 3).



Canvas from hungs of barrels embedded in nutrient solution after germination of spores. (Natural size.)

FIG. 3.

With regard to (a) it is difficult to explain the undue resistance of the mould spores to such great heat, and no reference or mention of such a high temperature could be found in the literature. It is to be noted, however, that the oil is heated under dry conditions and under reduced pressure, both factors being usually additive so far as resistance of the spores to heat is concerned.

3. Leaving now the question of sterility of oils and fats and the containers, there remain to be considered the parchment paper and wrappers used for the finished margarine or butter. Many tests were made on all kinds and makes of paper, and in not a single instance was the paper found to be sterile, usually much the opposite. Mould spores, chiefly *Penicillium* and *Cladosporium* species, and various bacteria organisms abound. Figs. 4 and 5 show two tests made on different grades of paper. When such paper is used on margarine or butter the necessary dampness and nidus for the propagation of the organism is at once supplied. It is then only a question of time before dark green or black spots (colonies) make their appearance, ruining the butter or margarine as a saleable article.

Throughout the work, lasting nearly two years, the utmost precautions were taken to ensure freedom from contamination, either in drawing the samples or in making the tests; in many cases a sterile chamber was used when the sample required to be opened, even momentarily. One important point resulting from these investigations has had to be omitted for commercial reasons, viz., the ques-

tion of actually preparing a perfectly sterile oil without injuring its flavour or neutrality in any way.

Nothing but the utmost care and bacteriological cleanliness and sterile materials can help the margarine makers to put out a good, regular, butter-like product, which will keep its clean taste for a

It has been shown that many naturally occurring fats, particularly those of animal origin, may serve as valuable sources of one of these indispensable, but as yet unidentified, dietary components, the so-called fat-soluble A or vitamin A.

Our earlier researches led us to believe that oils of vegetable origin are practically devoid of this



FIG. 4.



FIG. 5.

Parchment wrappings embedded in nutrient solution, showing colonies grown from the mould spores existing in the paper. ( $\times 1$ .)

reasonable time. There is no doubt that failure to realise these conditions is largely responsible, not only for the tendency to rancidify, but for the variations in quality of butter and margarine with which we are so familiar.

Laboratory,  
The Craigmillar Creamery Co., Ltd.,  
Craigmillar.

## London Section.

Meeting held at Burlington House on March 7, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

### FACTORS INFLUENCING THE NUTRITIVE VALUE OF LARD AND LARD SUBSTITUTES.

BY J. C. DRUMMOND, D.SC., F.I.C.

Until comparatively recently the food value of oils and fats was regarded almost entirely from the calorific standpoint, and accordingly most of the experimental work was devoted to determining what percentage of the total calorific value of each product could be absorbed by the digestive system of the mammalian organism. The discovery of the vitamins\* by Hopkins in 1912, and the subsequent differentiation by later workers of three individual substances of this type has, however, led to a very fundamental change in our conceptions of the rôle of fats in the diet.

\* It is proposed to employ the term "vitamins" for these substances of unknown character rather than "vitamines" proposed by Funk, since the latter word implies, according to the Chemical Society's system of nomenclature, that the substances are of a basic nature. There is no evidence that this is so and they should therefore be classified as substances of unknown constitution (see Drummond, *Biochem. J.*, 1920, 14, 660).

valuable component, whereas the majority of animal oils and fats show its presence. (Earlier literature given by Halliburton and Drummond, *J. Physiol.*, 1917, 51, 235.)

As a result of improvements in the technique for conducting tests for the presence of the vitamin A, this view has been somewhat modified in a recent paper (Drummond and Coward, *Biochem. J.*, 1920, 14, 668), where it is shown that there is no hard and fast line between the vegetable and animal oils and fats when they are regarded from the standpoint of vitamin content, and that there are many factors which may influence the amount of this substance present.

In this paper it was pointed out that the chief factor determining the presence of the vitamin in animal products is the diet which the animal has been receiving. Apparently the vitamin A cannot be synthesised by the animal body, so that only when the diet contains ample supplies of this constituent can appreciable amounts be demonstrated in the tissue fats.

Quite early in the study of the vitamin present in certain edible oils it was observed that lard appeared to be devoid of this important constituent (McCollum and Davis, *J. Biol. Chem.*, 1913, 15, 167), and on many occasions subsequently this observation was confirmed by other investigators.

In a recent summary of the knowledge concerning the vitamins, the author wrote "no entirely satisfactory explanation of the deficiency of lard has yet been advanced" (Special Report No. 38, Medical Research Council. H.M. Stationery Office, 1919). Researches carried out since this statement was made have, however, provided what is regarded as a satisfactory solution to the question.

Before proceeding to describe the investigations on the food value of lard and lard substitutes from the point of view of vitamins, reference may be made to a few experiments which the author made some time ago on the digestibility of these products.

*The digestibility of lard and lard substitutes.*

The majority of edible oils and fats are well digested and absorbed by the mammalian alimentary tract, and it is very unusual to encounter a low coefficient of digestibility. There is a general opinion that the chief factor governing this coefficient is the melting point of the fats, and to a certain extent this appears to be correct, but it is doubtful whether the question is quite as simple as this. In the author's experiments all the samples of lard and of substitute lards which were investigated gave a high coefficient of digestibility.

TABLE 1.

Sample.	Approx. m.p. ° C.	Length of trial Days.	Average coefficient of digestibility. %
Neutral lard I. . . . .	37.5°	10	97.7
" II. . . . .	37°	10	98.2
Hardened cottonseed oil, lard substitute I. . . . .	40°	7	96.1
Hardened oil, lard sub- stitute II. . . . .	38°	10	97.2
Lard substitute A (com- pound lard) . . . . .	36°	7	96.4
Lard substitute B (com- pound lard) . . . . .	37°	7	96.7
Lard substitute C . . . . .	37°	7	97.5

These results confirm records by other investigators. On the basis of the old theories regarding the food value of fats, therefore, there would be little to choose between any of these products.

The newer knowledge of the vitamin content of oils and fats has taught us how erroneous such a conclusion may be, and accordingly a study of the vitamin content of the lards and lard substitutes was initiated. The technique of conducting these feeding tests is now so much improved that results of a roughly quantitative nature may be obtained provided very great care is taken (see Drummond and Coward, *Biochem. J.*, 1920, 14, 661). This biological method of testing is of course necessary in the absence of any chemical or physical means of estimation.

*The vitamin content of lard.*

Recent researches have demonstrated that the amount of vitamin A present in the body fat of an animal is determined to a great extent by the diet which the animal has received.

Apparently the animal organism does not possess the power to synthesise this peculiar substance, but can store up a reserve in certain localities such as the body fat when a surplus is provided in the diet. There did not therefore appear to be any reason why the pig should differ from other species in this respect. In collaboration with Capt. J. Golding, D.S.O., of the Research Institute for Dairying, University College, Reading, and Dr. S. S. Zilva, of the Lister Institute, experiments were made to throw light on these points.

At the outset it was evident to us that the usual dietaries on which pigs are raised in this country are in the majority of cases low in vitamin A. It therefore appeared quite feasible to argue that the growth of the pigs might demand most or all of the accessory factor in the diet, leaving little or no surplus for storage in the fatty tissue.

To test this point experimentally batches of young pigs were reared on dietaries some of which were practically devoid of the vitamin A, whilst others supplied ample amounts of that substance. These experiments have been fully described in a previous communication (Drummond, Golding, Zilva, and Coward, *Biochem. J.*, 1920, 14, 742).

Certain animals from each lot were slaughtered after several months on the special diets when

they had reached the size of porkers (about 180 lb.), and samples of leaf fat (peri-nephritic fat) and back fat were removed. By means of the biological method these fats were tested for the presence of the vitamin A, and the results proved to be most interesting.

Briefly this method of testing is carried out as follows:—Young animals, usually rats (since many standard data have now been collected for this species), are fed upon an artificial dietary which has been rendered as free from vitamin A as is possible. Upon this diet they are usually able to grow for a short time, presumably whilst they are utilising the reserves present in their own tissues, but after the elapse of a few weeks this growth ceases and the health and weight of the animals decline.

If at this point the diet is supplemented with the foodstuff to be tested the resulting effect on the health and growth of the animal is in the majority of cases dependent on the amount of the vitamin in the added substance. If no vitamin is present the decline of the young animals proceeds to a fatal termination, but on the other hand a recovery of health and a resumption of growth will indicate the presence of the accessory substance A. With the improvements in technique it is now possible to obtain roughly quantitative data by this method, but it is much to be hoped that a chemical or physical method will soon be available so that a much greater degree of accuracy will be attainable.

By this method the samples of fats removed from the various hatches of pigs were tested, and it was found that the body fats of swine fed on dietaries supplying ample supplies of the vitamin A contained that substance, but that its presence could not be demonstrated in the fats derived from the animals fed on the foods deficient in vitamin A.

It was therefore apparent to us that the pig resembles other species concerning which data are available in possessing the power to store supplies of the vitamin A in its body fat under suitable conditions.

The next step in the investigation was to ascertain why the majority of samples of lard contain no detectable trace of the accessory constituent. We were fortunate in obtaining the co-operation of Messrs. Harris and Co., of Calne, Wiltshire, at this stage of the work, and must express our appreciation of the very valuable help which they so readily rendered. From material which they placed at our disposal it was possible to show that the average samples of pig fat used in lard manufacture in this country contain small but appreciable amounts of the vitamin A.

The processes of lard manufacture were examined next in order to ascertain whether destruction of the vitamin occurs.

The destruction of the vitamin A at high temperatures was at one time believed to be due to the heating alone (Steenbock, Boutwell and Kent, *J. Biol. Chem.*, 1918, 35, 577; Drummond, *Biochem. J.*, 1919, 13, 81), but more recently it has been shown that temperatures up to 120° C. do not inactivate the vitamin unless there is contact with air or oxygen. It would appear, therefore, that the destruction is probably due to changes of an oxidative nature (Hopkins, *Biochem. J.*, 1920, 14, 725; Drummond, *Biochem. J.*, 1920, 14, 734; Zilva, *Biochem. J.*, 1920, 14, 740).

The older methods of lard manufacture, which still exist in many places, are essentially modifications of the original farmhouse process in which the fat is rendered at low temperature and separated in a simple manner from the connective tissue and water which forms the lower layer of the melted material.

Such a process would not bring about much oxidation, and accordingly it appeared probable that lards prepared in this manner would retain most of the vitamin present in the original fat.

Products of this type are usually termed "hutter's lard" or "home rendered lard."

This we have found to be true by actual experiments. Good recovery curves were shown by animals fed on a sample of pig fat and also on a lard prepared from this fat by a very simple rendering process in which the minced fat was melted in a steam jacketed pan at a temperature of approximately 70° C. The melted fat is stirred very little beyond what is necessary to obtain a satisfactory separation of the layers by adding salt, etc., and the clear, upper layer is merely run off and packed after being subjected to a simple straining or filtration.

Such a product appears to be as rich a source of the vitamin as the original crude fat. Very little lard is prepared by this type of process in Great Britain, but a similar method appears to be used extensively in America for preparing the products known as "Neutral lards Nos. 1 and 2" (Lewkowitzsch, 5th Edn., 1914, Vol. 2, p. 689). It is therefore rather surprising that American observers have not frequently encountered samples of lard containing the vitamin. Only quite recently has attention been drawn to such a product by Daniels and Loughlin (J. Biol. Chem., 1920, 42, 359) in the United States, but they advanced no explanation of their results.

It would seem probable, since these investigators took considerable trouble to check sources of error in their experiments, that they were working with lard prepared by a simple rendering process from pig fat containing comparatively a large quantity of the vitamin.

By far the largest proportion of lard made in this country is prepared by processes which involve a certain amount of aeration at high temperatures. Stirring takes place during the melting, and after the separation of the layers the fat is stirred at a temperature of about 102° C. for some 10–15 minutes to remove moisture. The dry fat is then frequently passed through filter presses before packing.

An active sample of pig fat was found to have lost practically the whole of its vitamin A after being converted into lard by such a process. It therefore appears probable that any technical process involving the contact of the hot fat with air or oxidising agents used for bleaching purposes will considerably lower the nutritive value of the product. The effect of filtration has not been investigated.

#### Lard substitutes.

(a) *Compound lards.*—Two products of this type were examined in an earlier investigation (Halliburton and Drummond, J. Physiol., 1917, 51, 235) and were found to contain no detectable traces of the vitamin A. Naturally the presence or absence of the vitamin will depend on the nature of the constituent fats.

Both the products investigated were composed mainly of cottonseed oil and oleo-stearin. Experiments on cottonseed oil show that the highly refined edible oil is usually deficient in the vitamin, but nothing is yet known as to the value of the crude oil or of the possible changes which may occur in the vitamin content during the refining processes. These questions are now being systematically studied in collaboration with Dr. Zilva.

Oleo-stearin also appears to be generally deficient in vitamin A. Beef fat normally contains appreciable quantities of this indispensable dietary constituent (Osborne and Mendel, J. Biol. Chem., 1915, 24, 379), and these observers found on crystallization of beef fat from alcohol that the vitamin is concentrated in the mother liquors together with the glycerides of lower m.p. This explains why in the process of separating oleo-stearin from premier

jus the greater part of the vitamin passes through with the expressed oleo oil. The usual process of oleo oil preparation would hardly be likely to affect the amount of vitamin present.

Considerable variations in the vitamin content of oleo oil may be encountered, but these are in all probability due to the seasonal variations in the diets of the animals. Oleo-stearin is practically always of very low vitamin value. The nutritive value of compound lards will therefore be much influenced by the vitamin content of the constituents employed in its compounding.

(b) *Lard substitutes prepared from hardened fats.* In a recent publication it was shown that a sample of whale oil which possessed a fairly high value as a source of the vitamin A was rendered completely inactive by being "hardened" at high temperatures (Halliburton, Paton, Drummond, and others, J. Physiol., 1919, 52, 325; also Drummond, Biochem. J., 1919, 13, 81). This was at the time attributed to the high temperature at which hydrogenation was carried out, but this opinion must now be modified in view of the more recent work on the stability of the vitamin to high temperatures in the absence of air or oxidising agents. The cause of the inactivity of the hardened fats from the vitamin standpoint has not been definitely ascertained, but a number of such products, both of animal and vegetable origin, have been examined in this laboratory recently and have shown no trace of the vitamin.

It is therefore not surprising that we have found several samples of lard substitutes prepared on a basis of hardened fats to be devoid of the growth-promoting factor, an experience which has also been encountered by investigators in America (see McCollum, Simmonds, and Pitz, Amer. J. Physiol., 1916, 41, 361).

#### Summary.

1. The food value of lard and lard substitutes cannot be estimated solely in terms of digestibility and coefficients of utilisation. Such products are usually well absorbed by the alimentary tract.
2. The nutritive value of lards and lard substitutes may be largely determined by the amount of the so-called vitamin A present.
3. The diet of the pig is the chief factor determining the amount of the vitamin present in the body fat.
4. In the manufacture of lard from a pig fat rich in the vitamin avoidance of aeration or close contact with air or oxidising agents at high temperature will prevent loss of the active substance.
5. Compound lards do not usually contain any appreciable amount of the vitamin A. This is because the constituents most frequently employed (e.g., cottonseed oil, ground nut oil, and oleo-stearin) are almost devoid of that substance.
6. The vitamin content of beef fat is dependent on the diet of the animal. In the manufacture of oleo-stearin from premier jus the vitamin passes through with the lower m.p. fractions into the oleo oil.
7. Samples of hydrogenated fats of animal and vegetable origin were found to be inactive from the standpoint of vitamin A, as also were samples of lard substitutes prepared on a basis of such fats. The cause of the destruction when it occurs has not yet been ascertained.

#### DISCUSSION.

The CHAIRMAN said that the results put forward by the author were of great importance, and tended to show the strong instinct which mankind had for consuming natural fats where possible. Work of this kind showed that there was something more than flavour which determined the desirability of eating butter in preference to margarine and similar artificial products.

Mr. E. R. BOLTON said that some twenty years ago it was an achievement to produce absolutely tasteless edible vegetable oils. Then the bacteriologist, by suitable cultures of bacteria, had introduced butter flavour into the fat, and now the biologist showed us that the presence of vitamins was necessary to perfect the oil for food. It would be desirable to find a method whereby the manufacturer could first separate the vitamins from oils, then carry on the refining process, and re-introduce the vitamins which otherwise would be destroyed by the process. It would be interesting to have experimental evidence as to how far the vitamins were destroyed by the process of heating necessary to produce clarification, and whether it was really exposure to oxygen, or heat, or both together, which had the detrimental effect. The process of refining oils in this country necessitated the chemical removal of the free fatty acids, followed by treatment at a very high temperature, but there was little or no exposure to oxygen. If, therefore, vitamins were destroyed in this process the destruction was not, in his opinion, due to oxidation. Olive oils were not usually exposed to high temperatures or to oxidation, and it would be interesting to have a comparison of the vitamin content in edible olive oil and in refined oils.

Dr. G. W. MONIER-WILLIAMS said that one interesting point was the very high vitamin content of cod-liver oil and whale oils. Terrestrial animals apparently derived their vitamins from vegetable sources. The vitamin content of butter fat was higher in the spring when the cows were grass-fed than in the winter when they were largely stall-fed. From what sources did the whale and the cod obtain their store of vitamin? Presumably it must originate with the marine "plankton," the unicellular organisms which formed the ultimate food material of all marine animals. If so, it would be interesting to trace the course of these vitamins from the organisms in which they were originally formed, through the various marine animals, until they were ultimately deposited in such relatively large amounts in cod-liver and whale oils.

Mr. H. F. E. HULTON asked whether the lard had been given to the rats in the natural state or cooked. If previously fried the vitamin content might be very much reduced, and he thought the rat experiments should therefore be carried out with lard as usually consumed, since, unlike butter, it was not eaten as bought.

Mr. T. THORNLEY asked if hydrogenation was the only process to which the whale oil had been subjected. It was difficult to hydrogenate whale oil without some previous refining on account of catalyst poisons being present in the whale oil, and it might have happened that some of the refining processes had destroyed the vitamins. He further asked if simple hydrogenation of an unrefined oil at a low temperature had been shown to destroy vitamins, in view of Sekine's work showing that the vitamin content of commercial cod-liver oil, poor in vitamins, was increased by hydrogenation at a low temperature.

Captain GOLDING said that his share in these experiments had been in the pig-feeding trials. Not only was there a great rise in the curve of growth of the animal when a sow was given cod-liver oil after it had practically ceased to grow on a diet deficient in fat-soluble A, but there was also an improvement in the general well-being of the sow after the change of diet which figures fail to indicate. Crude cod-liver oil was not particularly palatable, but the pigs took it readily. Pigs which had been kept on a diet deficient in fat-soluble A would endeavour to make good the deficiency by eating grass. These observations showed that the question of taste, to which the Chairman

had referred, was really a very natural guide, particularly in the animal world.

Mr. A. E. PARKES inquired whether the process of curing pork for conversion into bacon had any effect on the vitamin content.

Dr. DRUMMOND, in reply, said that the refining and deodorisation of oils were part of the investigations now in hand. It was realised that the only way to get reliable information was to take the oils from the starting point and follow them through every stage of manufacture, testing them at each stage. Every such test took some three months to complete, and might necessitate the employment of two or three dozen animals, so that it was a tedious and expensive method. The questions of refining, the removal of the fatty acids, bleaching and filtering through various materials, deodorising, high-pressure steaming, etc., were being worked out. With regard to olive oil, he had not yet made controlled investigations, although in one case he had found evidence of a certain amount of vitamin A in a sample of crude olive oil. His information as regards this oil, however, was scanty. He agreed with Dr. Monier-Williams that it was extraordinary how the cod was able to store such a large amount of vitamins in its liver. The chief types of fish of the white variety—the cod type—appeared to store large quantities of vitamins in the liver, but in the other type of fish, the herring type, the vitamins appeared to accompany the fat distributed in the muscle tissue. He believed that in the case of the cod and the whale, the vitamin was derived primarily from simple marine organisms, and he hoped to go to north Norway, with some colleagues, to find out how the cod obtained its vitamins. It might be that the amount of vitamin produced in marine plants was far higher in proportion than with the ordinary land plant. An eminent marine biologist was accompanying the trip to Norway to throw light on this aspect of the problem. The question of summer and winter butter had been worked out in the preliminary stage, and it had been found that grass-fed cattle in the summer gave milk which possessed a higher vitamin-content than milk from cattle during the winter when the feeding was stall-feeding, such as cake, etc. With regard to the cooking of lard and the experiments on the rats, the experiments were carried out not so much to find out the food value of the fat as consumed, but to settle whether the pig could store vitamin in its fat, and how the vitamin was affected by the technical processes of extraction and refining. Further experiments were in progress. He had tested a sample of crude whale oil before and after hydrogenation. It was possible that the oil had also been refined, but as yet there was no knowledge of the effect of these processes, particularly of the removal of the free fatty acids. Low-temperature work had not yet been carried out. He had not seen the paper referred to by Mr. Thornley, but if the destruction actually occurred by oxidation, it was quite feasible that the vitamin might, perhaps, be restored by a corresponding reduction process.

## THE STABILITY OF BENZOYL PEROXIDE.

BY ROBERT CROSSIE FARMER.

Benzoyle peroxide is of industrial interest as an oxidising agent, and has been utilised in several directions under the name of Lucidol. It was introduced by the Verein, Chem. Werke of Charlottenburg as a fixing agent in technical microscopy. It is a powerful antiseptic (Loevenhart, Bull. Comm., 33, 471; Year Book of Pharmacy, 1906, 95), and has been used for the treatment of burns and ulcers. It has a slight anæsthetic action when applied externally. Its internal use has been



given up on account of its poisonous action on the blood (Fränkel, "Die Arzneimittelsynthese"). Its physiological action on dogs was examined by Nencki and Zaleski (Z. physiol. Chem., 1898, 27, 487).

Benzoyl peroxide acts as a drier towards linseed and other drying oils (Krumbhaar, Farben-Zeit., 1913, 18, 1280), and has some application for bleaching oils (G.P. 214,937. See also Utz, Chem. Rev. Fett- und Harz-Ind., 1912, 19, 70, 128, 151; Farben-Zeit., 1912, 17, 2105).

Benzoyl peroxide has a limited application for explosive purposes; since its temperature of explosion is low, its use for electrical detonators acting at low tension has been patented (E.P. 23,450 of 1912).

The compound has generally been assumed to be quite stable for handling and storage. Its behaviour on warming led the author to examine more closely into its stability, and the results given in the present paper indicate that some care should be taken when dealing with dry benzoyl peroxide.

For the preparation of benzoyl peroxide in larger quantities the autoxidation of benzaldehyde does not proceed readily, but the action of sodium peroxide on benzoyl chloride (Nencki and Zaleski, loc. cit.) was found satisfactory. The crude product melts at about 98° C., but on crystallisation from alcohol the pure compound of m.p. 103°–104° is readily obtained. It is practically insoluble in water, and almost insoluble in petrol, but dissolves readily in benzene and most organic solvents. It is gradually attacked by warm ammonia, forming benzamide and ammonium benzoate. Sodium hydroxide gradually dissolves it with formation of sodium benzoate.

Very little information has been published regarding the stability of benzoyl peroxide. It is known to decompose vigorously on melting in a capillary tube, and Orndorff and White found that molecular weight determinations at the boiling point of benzene were inaccurate owing to decomposition (Z. physik. Chem., 1893, 12, 68).

Tests made by the author at the Research Department of the Royal Arsenal, Woolwich, are given below by the permission of the Director of Artillery. The stability was measured by means of the velocity of evolution of gas in a vacuum, by the standard method used for testing high explosives, as described by the author (Chem. Soc. Trans., 1920, 117, 1432). The benzoyl peroxide used in these tests was of German origin, and showed the correct melting point for the pure substance. It will be seen that the decomposition proceeds with an acceleration, indicating autocatalysis. For comparison it may be stated that the main high explosives, such as picric acid and trinitrotoluene, show no measurable gas evolution at 100°, whilst tetryl gives only about 0.06 c.c. per g. in 100 hours.

#### Vacuum stability tests.

(i.) Test at 49° C. Benzoyl peroxide m.p. 103.5°–104° (5 g.) previously dried for 100 hours in a vacuum to remove all volatile matter.

Hours	0	60	100	150	200	250	300
c.c. of gas (corr.)	0	0.37	0.93	1.37	2.53	3.55	4.63

(ii.) Tests at 80°. Benzoyl peroxide as above (1 g.)

Hours	0	5	10	15	20	24
c.c. of gas (corr.) (a)	0	0.55	1.20	2.10	3.60	6.40
(b)	0	0.60	1.35	2.40	4.10	7.45

(iii.) Tests at 100°. Benzoyl peroxide as above. (a) 5 g. of peroxide exploded within 30 minutes, breaking the glass apparatus, but without injury to the metal bath. (b) 1 g. of peroxide was heated first to 80° in a vacuum and showed a similar rate of decomposition to the above tests at 80°, namely, 0.31 c.c. of gas in 3 hours. The temperature was

then raised to 100°, and explosion occurred within 1 hour.

This instability is confirmed by observations on the behaviour of benzoyl peroxide made at this laboratory, which may be summarised as follows:—

#### Tests at atmospheric pressure.

(i.) When 1 g. of the crude peroxide was heated at 100° a mild explosion occurred after 30 minutes, but the glass tube was not broken. (ii.) One gram of the peroxide, crystallised from alcohol, was heated at 100°. A mild explosion occurred after 1 hour; the glass tube was broken, but the bath was not damaged. Similar explosions were observed on heating pure benzoyl peroxide under water at 100°. (iii.) 7 g. of the crude peroxide was kept at ordinary temperature in a dish in a vacuum-desiccator over sulphuric acid. After 8 hours the peroxide exploded with a sharp noise and destroyed the desiccator. The remains of the peroxide formed a charred mass. (iv.) Slight explosions were observed on grinding dry benzoyl peroxide in a mortar.

The gas evolved on decomposition was carbon dioxide and amounted to approximately one molecule per molecule of benzoyl peroxide. In every case the decomposition was accompanied by an odour similar to that of phenyl benzoate.

**Safeguards against explosion.**—Wet benzoyl peroxide has shown no explosive tendency at ordinary temperatures, and the manufacture thereof appears to be without danger, so long as the compound is not allowed to become dry. In order to prepare a non-explosive dry product, salts were mixed with wet peroxide, and the mixtures dried at 50°. A mixture of 1 part of benzoyl peroxide and 4 parts of sodium chloride decomposed gradually without explosion when heated to 130°. When a flame was applied to a heap of the mixture, the peroxide burned out gradually, but on removing the flame the burning ceased. The benzoyl peroxide can be extracted with benzene from the salt mixture and gives the correct melting point. After keeping the mixture at 45°–50° C. for 20 days, the benzoyl peroxide on extraction still gave approximately the correct melting point. Experiments with ammonium phosphate gave similar results, but the sodium chloride mixture appeared less inflammable of the two.

**Summary.**—Benzoyl peroxide has explosive properties in the dry state, and drying should be avoided, where possible, in the manufacture. This instability applies both to the crude and the pure compound. The peroxide can be rendered immune against explosion by mixing it in the wet condition with excess of salt and drying the mixture. On warming, the substance decomposes gradually with evolution of gas, and the decomposition is autocatalytically accelerated. Storage of the peroxide under warm conditions is therefore to be avoided.

Research Laboratories,

W. J. Bush and Co., Ltd.,  
Hackney.

#### DISCUSSION.

Dr. H. P. STEVENS asked the author how far purity affected stability. Some time ago he had made some experiments with benzoyl peroxide for vulcanising rubber, but he had not taken any particular care in purifying it. A small quantity had been left standing in a watch glass in a dry, warm place 4 or 5 feet above a gas stove, and it had exploded without being touched. He had mixed benzoyl peroxide with rubber without difficulty by keeping it wet. He wondered whether a carefully purified product would be more stable.

Dr. M. O. FORSTER asked if it exploded when hammered.

Dr. FARMER replied that he had not given it very vigorous hammering. Purity did not, in his experience, affect the stability particularly. He had had the material in a state of great purity, and also in various degrees of impurity, but the time that it would stand beating seemed to be about the same. It did not seem to undergo any increase in stability on purifying. In the case mentioned by Dr. Stevens, possibly the benzoyl peroxide had become very dry by the gradual beating, and so had become more sensitive to heat.

Dr. STEVENS said that in his case the benzoyl peroxide had been mixed on warm rollers heated to 50° or 60° C., and a little spot of benzoyl peroxide had got on this roller and dried, and exploded without any friction.

## Newcastle Section.

Meeting held at Middlesbrough on March 7, 1921.

DR. J. H. PATERSON IN THE CHAIR.

### THE MANUFACTURE OF ALCOHOL FROM COKE-OVEN GAS.

BY C. F. TIDMAN.

The conversion of the ethylene present in coke-oven gas into alcohol and its derivatives was dealt with by my late chief, Mr. Ernest Bury, at a meeting of the Cleveland Institution of Engineers in December, 1919 (see J., 1920, 94A). The present paper contains a record of the work that has been carried on during the last twelve months on this subject. The suitability of alcohol as a fuel has already been established, and some figures which will be given in a later part of this paper will indicate how far these claims will hold when the power alcohol has been obtained synthetically from coke-oven gas.

Berthelot (1854) is generally credited with the discovery that ethylene could be fixed as ethylsulphuric acid, which can then be hydrolysed to alcohol, although Henry Hennell indicates in a paper communicated to the Royal Society in 1827 that he had identified sulphovinic acid in quantity of sulphuric acid given him by Faraday, which had absorbed eighty times its volume of olefiant gas, or, in other words, 5% of its weight of ethylene. In a paper published the following year he states that this sulphovinic acid decomposed into sulphuric acid and alcohol. Fritzsche seems to have investigated in 1897, and more thoroughly in 1912, the application of this synthesis to industry, but when he found that 12 cubic metres of scrubbing surface was required per cub. m. of gas treated per minute he decided that the large scrubbing plant necessary would render the proposal unworkable. In the work we have conducted at Skinninggrove we have decided that not only surface, but turbulent contact plays a part in deciding the speed with which ethylene will combine with sulphuric acid. While Fritzsche seems to have worked along the lines of pressure, we have considered temperature and turbulence to be essential factors in successful and rapid absorption. Since the publication of Mr. Bury's work on this subject, a patent has been taken out by a Belgian firm, where success seems to have been attained by the use of catalysts such as vanadic and tungstic acids.

With very few data at our disposal the problem was attacked at Skinninggrove under the direction of Messrs. Bury and Ollander. The first point was to determine the most suitable temperature of

absorption, and to this end absorptions were carried out at temperatures 160°–165°, 95°–100°, and 85°–90° C. respectively. The absorption apparatus consisted of two lead pots in series provided with stirring gear and containing 95% R.O.V., through which the coke-oven gas was passed in series. At 85°–90° C. it was found that there was 0.25% of carbon in the acid, showing that even at this temperature decomposition was occurring, while at 160°–165° C. the acid used soon became gelatinous, while the exit gas contained a good deal of sulphur dioxide. Fig. 1 shows the progress of the absorption at temperatures below 85°. The figures for

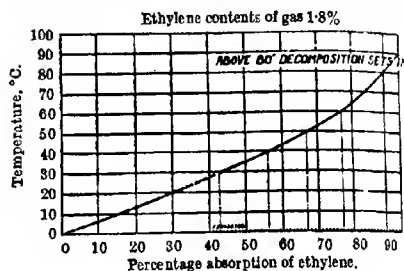


FIG. 1.

Variation of absorption of ethylene with temperature.

these absorptions were obtained by using towers packed with fused silica as absorbers. From these data the best temperature of absorption was considered to be from 60° to 80° C. Four lead towers, each 4 ft. 6 in. by 3 in. diam., jacketed by a cylinder 5 in. diameter, arranged in series, were packed with broken fused silica in such a way that the ratio of free space to packing space was as 2:5. The total capacity of the towers was 1 cb. ft., of which 5/7 cb. ft. represented solid packing material. Sulphuric acid containing 95.4%  $H_2SO_4$  was fed down the towers by means of a siphon and carried to the top at intervals. During the first few experiments acid was fed down the tower continuously in the smallest continuous stream possible (at the rate of about 30 c.c. per minute), but later experiments showed that this was not necessary, since after flushing down the towers the acid flow could be stopped and the absorption continued without appreciable alteration for an hour. In one experiment, for example, the inlet gas contained 1.70% of ethylene; after 5 minutes' absorption the outlet gas was found to contain 0.60% of ethylene. The acid flow was then stopped and the percentages of ethylene in the exit gases were found to be as follows: 10 min., 0.60%; 15 min., 0.64%; 20 min., 0.64%; 60 min., 0.66%. A repeat experiment gave almost identical figures.

In the first absorption experiment carried out, desulphurised gas was passed through a bubbler containing 80% sulphuric acid. The primary object of this scrubber was to remove moisture and any higher homologues of ethylene (propylene, butylene, etc.). In the gas produced under the conditions existing at Skinninggrove, however, the presence of the higher homologues has not been observed, although doubtless these exist in small quantities. During the course of an experiment in which a gas richer in ethylene than coke-oven gas was used—viz., gas driven off from beech-wood charcoal, which had been placed in the gas stream—a white, crystalline solid separated out on the 80% acid; this was found to have the empirical formula  $C_4H_8$ , m.p. 128° C., b.p. 250°–255° C. With this solid was associated a second solid containing oxygen and of the probable empirical formula  $C_4H_8O$ ; it had a very characteristic sweet taste which persisted in the mouth for hours. Even

using ordinary coke-oven gas a certain amount of solids settles out in the preliminary hoppers, but these are easily separated and can be discarded, although further investigation may prove them to be of value.

After the gas had left the scrubber containing 80% acid it was passed through the experimental towers just referred to at the rate of 5–6 ch. ft. per hour, and at the commencement the absorption was as high as 90% of the ethylene entering the system. The experiment was made a continuous one and lasted 145 hours. The average speed of the first 440 ch. ft. of gas was 5.6 ch. ft. per hour, while the average absorption of the ethylene was 90%. The speed of the next 320 ch. ft. was 4.8 ch. ft. per hour, with an average absorption of 85%. The total quantity of acid (95.4%  $H_2SO_4$ ) used was 3000 c.c. With a gas space in the towers of 2.7 ch. ft. and a gas speed of 5.6 ch. ft. an hour, the time of contact of gas with acid was thus 3.1 minutes, during which 90% of the ethylene present was absorbed. These data multiplied up to plant size, where the daily carbonisation is 800 tons, would suggest the use of nine scrubbing towers, each the size of the ordinary benzol scrubber, i.e., 10 ft. diam. and 80 ft. high. Of these nine towers practically the volume of six would be occupied by packing material, the function of which is to give surface and turbulence. To give surface alone, special packing material was used at Skinningrove in plant experimental towers, but although a large surface was provided, allowing three minutes' contact, 80–90% absorption of the ethylene was not obtained.

Keeping in mind these facts, a series of experiments was carried out in a flask of 1950 c.c. capacity filled with dry desulphurised coke-oven gas. The flask was heated in a water-bath until the temperature of the gas was about 80° C., hot acid (300 c.c.) was added and the flask covered with a non-conducting material and vigorously agitated, to afford adequate surface contact, for periods varying from  $\frac{1}{2}$  to 3 minutes. Further experiments were carried out using 5% of silica with the 95.4% sulphuric acid. The results obtained are shown in Fig. 2, from which it is seen that the addi-

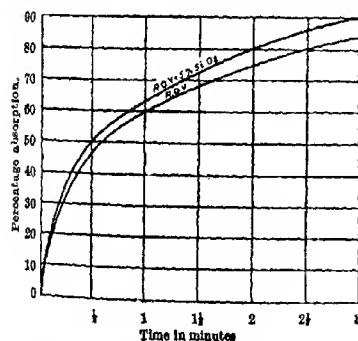


FIG. 2.

Speed of absorption 80° C. 95.4%  $H_2SO_4$ .

tion of silica increases the absorption by approximately 5%. The function of the silica is not quite clear, but it appears to have a catalytic action and not merely a surface effect, since during the first few experiments on the small laboratory towers the absorption was particularly good, while a much later experiment, using the same towers, showed a falling off in percentage absorption, conditions remaining exactly the same except that the silica packing was coated with a slime of lead sulphate. In the experiments just described the addition of

silica greatly increased the amount of froth formed on the surface of the acid, this giving much better contact between gas and acid.

From the figures quoted it will be seen that for 3 minutes' contact by either method—towers or direct agitation in flask—the percentage absorption is 80%, so that whichever method of scrubbing is finally adopted—i.e., towers or mechanical washer—it should be possible to remove 70–80% of the ethylene by allowing 2½–3 minutes' contact.

Spraying the acid into the gas has also been tried, but difficulties occurred in maintaining the required temperature and also in the blocking of the sprays. It was decided that this type of scrubber was not suitable. However, certain forms of mechanical washers give practically the spray effect without the disadvantages of blocking of small jets as used in sprays.

The next point to be determined is how nearly sulphuric acid can be saturated with ethylene before it will cease to act as an efficient absorber. Mr. Bury stated that 18% of the acid radicle is available for ethylene absorption, i.e., 95% sulphuric acid (R.O.V.) would continue to absorb ethylene until reduced to 77%  $H_2SO_4$ . We have since found, however, that acid already saturated 18% would still continue to absorb ethylene in a mechanical agitator, 30% of the ethylene present being absorbed in 2½ minutes.

A mechanical washer would have the advantage over a tower scrubber system in that the size of the plant would be much reduced, and the power required to drive such washers is not great, considering the power required for pumping and the inconvenience of periodically cleaning a tower scrubber. The absorption product contains free alcohol in addition to sulphuric acid, ethyl hydrogen sulphate, and diethyl sulphate. This was apparent from the results of a trial run on the experimental plant. From analyses of the gases from each scrubber it was seen that No. 1 scrubber gave a shortage when distilled for alcohol, but scrubbers 3 and 4 each yielded more alcohol than was indicated by the gas test. In a number of tests the free alcohol has been as high as 50% of the total yield. It was therefore decided to recover the alcohol by distillation under reduced pressure, first allowing distillation to proceed without steam, and admitting steam when the temperature of distillation increased so that the speed of distillation was maintained. The following figures indicate the procedure. The acid was first diluted to 84%  $H_2SO_4$  by the addition of water. Under a vacuum of 26½ in. distillation commenced at 94° C., the highest temperature recorded being 146°. The alcohol distilled over satisfactorily, and no frothing took place. The finished acid, which was free from alcohol, free or combined, showed 80%  $H_2SO_4$ .

The method suggested earlier for recovery of the alcohol was to use a counter-current flow of acid and superheated steam. Over short periods this method was satisfactory, but after a time large amounts of froth were produced which passed into the condensing system.

The bulk of the alcohol obtained from this process is in the form of a 15% distillate, any weaker distillate obtained being used for diluting the acid to the strength required for distillation in vacuo. The production of strong alcohol from this weak distillate is hardly within the scope of this paper, since the distillate is of a very similar strength to that obtained by fermentation. The alcohol itself, however, is very interesting in view of the impurities present. These are not important when the alcohol is to be used as fuel, since the alcohol burns completely—indeed, it has been considered possible that the Revenue authorities may even accept the spirit as sufficiently denatured to pass into use without the addition of other substances; certainly the crude spirit is much less potable than

methylated spirit. The alcohol, however, possesses an objectionable odour, which persists in other products made from it. For example, chloroform made from by-product alcohol was reported to satisfy B.P. requirements only after treatment with hot concentrated sulphuric acid to destroy the odorous substances, the washing process reducing the bulk 10%. In preparing strong alcohol from the weak distillate it had been noted that the weak distillate was often cloudy but became clear on heating, showing that the substance present was more soluble in hot than in cold alcohol. Repeated distillations from the 80% alcohol left a residue which on cooling deposited a white, crystalline solid, which was found to be diethylene disulphide (m.p.  $111^{\circ}\text{C}$ ., uncorr.). In addition to this impurity a yellow oil containing about 15% of sulphur was found on the surface of the weak distillate. Both forms of impurity were present in largest amount in the first scrubber. Since only a small quantity of the oil is available and it has a wide range of boiling point, it has not been possible to fix definitely the exact substance present. A further difficulty in that diethylene disulphide is very soluble in this oil and consequently it is difficult to obtain the oil free from this substance.

In using an 87% alcohol as fuel without any attempt to remove impurities, quite good results have been obtained in a number of practical trials. In one trial with a 4 h.p. Triumph machine (total weight just over 700 lb.) the following results were obtained:—No. 1 Shell petrol, 64.6 miles per gall.; 50% benzol, 50% alcohol, 78.50 miles; alcohol, 58 miles per gall. A trial with denatured alcohol made from coke-oven gas on a Buick car also gave satisfactory results. The presence of diethylene disulphide appeared to give rise to no bad results.

The diethylene disulphide is derived from the hydrogen sulphide in the coke-oven gas, which contains 6 g. of  $\text{H}_2\text{S}$  per cb. m. The gas for use in the small alcohol plant at Skinningrove is subjected to the ordinary bog iron-ore system of purification. To instal such a system for dealing with 8 million ch. ft. of gas per day is out of the question, since both in capital outlay and labour the cost would be prohibitive. Again, it is very difficult to rid a gas entirely of sulphur compounds by using bog iron-ore purifiers. An alternative method has therefore been devised depending on the well-known reaction between hydrogen sulphide and sulphur dioxide. At first gaseous sulphur dioxide, obtained by the action of acid on sodium bisulphite, was admitted into the gas stream, and the mixture passed up a packed tower. By admitting rather less sulphur dioxide than was theoretically necessary to decompose the hydrogen sulphide it was hoped to precipitate the bulk of the sulphur in the tower, thus producing a gas containing only a small amount of undecomposed hydrogen sulphide which was to be removed by a bog ore purifier. In practice it was found that when the amount of sulphur dioxide present was short of that required very little decomposition took place. The admission of a small quantity of steam gave very little improvement, and passing the mixed gas through hot tubes also failed to remove the sulphur. It was therefore decided to try to desulphurise the gas by the counter-current system, using sulphur dioxide in solution. This method was found to be quite successful, the gas being easily purified when a large excess of sulphur dioxide was present. The exact excess necessary will no doubt depend largely on the plant itself. Much of the excess of sulphur dioxide is carried forward in the gas stream, but it can easily be removed by scrubbing with water. The sulphur produced in this process is deposited in the form of a mud which can be easily separated from the liquid.

Mr. Bury has pointed out that at a coking plant such as that at Skinningrove, with its 120 Otto regenerative ovens, the make per week, allowing 70% absorption and 70% recovery, would be 9280 gallons of absolute alcohol. When pure gas is used and the alcohol is recovered by distillation under reduced pressure we may easily expect an 80% recovery, while using an acid stronger than 95% R.O.V. we may expect the 70% absorption of the ethylene to be increased. It would not be difficult to obtain, say, 98%  $\text{H}_2\text{SO}_4$  if instead of erecting a sulphuric acid plant with an acid concentrating plant an oleum plant were installed. The 80% acid left after dealcoholation would form a very good absorbent for the  $\text{SO}_2$ , while the additional acid thus made would be used for benzol washing etc. The scheme at Skinningrove has been worked out using an acid of 95%  $\text{H}_2\text{SO}_4$ , i.e., R.O.V., but we have noted that an increase of 1 or 2% acidity is of advantage. When using acid of 93%  $\text{H}_2\text{SO}_4$ , we have found that the absorption falls off very rapidly. The figures given by Plant and Sidgwick (J., 1921, 14 r), using concentrated ethylene, show that while 93.1% acid increases in weight 20.8% on absorbing ethylene, 95.8%  $\text{H}_2\text{SO}_4$  increases 28%. At some point between 95.8% and 98.1% we get a decided increase in the amount of diethyl sulphate formed, while when free sulphuric anhydride is present carbonyl sulphate is formed—a substance not hydrolysable by alcohol. When using R.O.V. 55% (approximately) we have not found diethyl sulphate to be formed, although in certain of the crude alcohols produced on the experimental plant traces of the characteristic odour of diethyl sulphate have been noticed. We have also found it advisable to place one or two acid bubblers before the 80% sulphuric acid scrubber to avoid the formation of a substance like pitch, which may easily block the scrubber. When the bubblers are used this tarry product separates out on the surface of the acid and can be removed from time to time. We have not yet determined the composition of this deposit, but in a preliminary examination we have found that it is only partly soluble in water. The soluble portion gives a deep-green solution, and when freed from acid forms a soapy solution giving a persistent film when air is blown through it.

In conclusion, I desire to express my thanks for the sympathetic help and encouragement received from the heads of the Skinningrove Iron Company, Mr. Alfred Hutchison, and Mr. Coulson. I also wish to acknowledge the great help received from Mr. Ralph Robinson, B.Sc., Mr. G. M. Harrison, and Mr. K. L. Brown.

#### DISCUSSION.

The CHAIRMAN suggested the desirability of research into the possibility of using the hydrocarbons of coke-oven gas (which contained a total of 30%) as a source of fuel rather than selecting one small and unimportant constituent for conversion into fuel. The sulphur problem was an extremely serious one having regard to the effect of the sulphur compounds upon the fuel tanks and exhaust apparatus of an engine, and any process devised for the manufacture of power alcohol would have to produce a material practically free from sulphur compounds.

Dr. G. WETMAN thought that there should be no great trouble or expense involved in removing hydrogen sulphide, as gasworks were able to make a profit on oxide purification. Gasworks had necessarily to remove every trace of hydrogen sulphide, and there was no trouble in dealing with 10 or 20 million cb. ft. per day. He was not sure if oxide purification plant would not be less expensive than the scrubber system of removing excess of sulphur dioxide which Mr. Tidman had mentioned. In an older process the last trace of sul-

phur dioxide was removed in an alkaline washer. The reaction between sulphur dioxide and hydrogen sulphide was most interesting, but extremely complicated. There were other sulphur compounds besides hydrogen sulphide which might cause the trouble referred to.

Dr. J. T. DUNN suggested that if it were necessary to have the final product free from sulphur, attention would have to be given to the removal not alone of hydrogen sulphide, but of the other sulphur compounds which accompanied it in the gas.

Mr. A. TROWBRIDGE said that many years ago he had had considerable experience in separating the last traces of hydrogen sulphide from gas in connexion with the development of the Chance process for the recovery of sulphur from alkali waste. Large quantities of hydrogen sulphide had been passed through the Claus kilns, and it had been necessary to determine the exact proportion of air necessary to give the maximum yield of sulphur; it had been found impossible to obtain an exit gas free from either hydrogen sulphide or sulphur dioxide. The United Alkali Co. had had a similar experience recently at their Gateshead works. He thought that the use of sulphur dioxide for removing the hydrogen sulphide from coke-oven gas did not hold forth much promise, and that some other method should be devised.

Mr. J. E. CHRISTOPHER said that he had been in touch with a plant in which sulphur was recovered from waste gases, the best yield being obtained when the final gases showed signs of both  $H_2S$  and  $SO_2$ . If either were present alone the yield was diminished. He foresaw difficulty in introducing alcohol recovery apparatus in many existing coke plants, but the question should be considered more especially in regard to new plants. Had Mr. Tidman any experience of this particular process in dealing with gas from a low-temperature carbonisation scheme? A very high yield of motor spirit would be expected in that case, and if it could be established it would undoubtedly bring low-temperature schemes more into prominence.

Mr. A. HUTCHINSON said that the paper was really a report of progress in the conversion of a process which had proved a success on a small laboratory scale to a semi-manufacturing scale, with a view to finding the most economical plant for large-scale manufacturing, so as to keep down the capital expenditure to a minimum. So far, the experiments had proved entirely successful, but the number of towers required to complete the extraction of the whole of the ethylene by means of sulphuric acid was so considerable that they were now turning their attention to a small experimental plant designed by Dr. Davidson, which had been successful in ammonia absorption, and, it was hoped, would prove equally so in ethylene absorption. If this proved to be the case, a very considerable saving in capital cost would be effected, and there was every likelihood of the manufacture of alcohol being a paying proposition. He did not think that the sulphur compounds would have any detrimental effect in the use of the alcohol for internal combustion engines, motor cars, etc. Experience at Skinningrove with large gas engines using gas containing a certain amount of sulphur was that the engines were themselves in no way affected by the sulphur. When the waste gases were cooled in water, however, the acid solution affected the exhaust pipes.

Mr. H. CROWE said that at Skinningrove they had run their large gas engines on blast-furnace gas only, which contained only traces of sulphur. They had also for many years run these engines on a mixture of blast-furnace gas and coke-oven gas containing a considerable quantity of sulphur.

No ill-effects from the sulphur were felt in the engine until at the point, just past the exhaust valve, where water was injected to cool the exhaust pipes and deaden the noise of the exhaust. From this point onwards, all along the exhaust pipes and silencers, rapid corrosion took place. Large gas engines were more satisfactorily run on pure blast-furnace gas than on a mixture of blast-furnace gas and coke-oven gas.

Major SCOTT said that he did not think the action of sulphur on the interior of internal combustion engines would be at all serious. He had run gas engines on blast-furnace gas and gas from gas producers and had found no bad results in the cylinders. Corrosion was liable to take place in the exhaust pipes owing to moisture being present.

Mr. TIDMAN, in reply, said that after removing the diethylene disulphide from a sample of crude alcohol by means of mercuric chloride, the resultant spirit was pronounced by the visiting Excise officer to be equal to grain spirit. His work had all been carried out on gas produced under coking conditions as at Skinningrove, where metallurgical coke was the main product, and had not extended to low-temperature carbonisation. He suggested that such substances as thiophen would be removed in the wash oil of the benzol scrubbers, and so would not trouble the alcohol system. Replying to Mr. Trowbridge, Mr. Tidman said that when removing the hydrogen sulphide by means of sulphur dioxide solution in the experimental towers referred to, the gas from the exit of the last tower contained no sulphur dioxide.

## Communications.

### GUANIDINE CARBONATE AS A STANDARD ALKALI.

BY A. H. DODD, B.A., A.I.C.

Sodium carbonate is very generally used for the preparation of standard alkali solutions, but it is open to several objections (*cf.* Rimhach, *Ber.*, 26, 171; Higgins, *J.*, 1900, 958; North and Blakey, *J.*, 1905, 396).

During the last three years guanidine carbonate has been used in this laboratory for making up standard alkali solutions, and has been found to possess certain definite advantages over sodium carbonate. Thus it can easily be freed from inorganic impurities, it contains no water of crystallisation, and, when pure, it is non-hygroscopic.

No reference to its use as a standard reagent has been found in the literature, although Grossmann and Schuck (*Chem.-Zeit.*, 30, 1205) have suggested its determination by titration with standard acids and recommend it as a powerful alkali to precipitate zinc, etc.

Guanidine carbonate,  $[NH_2C(NH_2)]_2 \cdot H_2CO_3$ , is a strong "non-acid" base; the hypothetical guanidonium hydroxide from which it is derived approximates in strength to the alkali hydroxides, and is stronger than the tetra-substituted ammonium hydroxides (Ostwald, *J. prakt. Chem.*, (2), 33, 367). The heat of neutralisation of the free base is  $14.12^\circ C.$  per g.-mol. (Morrell and Bellars, *J. Chem. Soc.*, 1907, 1012).

The carbonate as ordinarily prepared from guanidine thiocyanate, i.e., from ammonium thiocyanate as starting material, is not to be recommended. Generally it is hygroscopic and contaminated with ammonium and potassium salts. In any case the purification is costly, and the yield of

carbonate from thiocyanate, using potassium carbonate and alcohol extraction, is only 70% (Volhard, J. prakt. Chem., (2), 9, 10).

The product made from dicyanodiamide, i.e. from calcium cyanamide as raw product, is extraordinarily pure, and should be obtainable for a few shillings a pound.

Guanidine carbonate is precipitated from its aqueous solution by addition of alcohol, and it is best to make use of this fact to obtain the analytical reagent. Two precipitations from its aqueous solution and washing with 80% alcohol yield small white crystals which, after drying at 110° C., show a purity of 100% if tested with standard acids, and can be stored in a well-stoppered bottle and used without further treatment for standard alkali for works use. Samples left covered but exposed to the atmosphere for 14 days in a warm corner of the laboratory did not gain in weight appreciably. Beautiful colourless crystals weighing up to 3 g. can be obtained by slow evaporation from water, but although they are anhydrous they would not, however, be used for analytical work, since they are liable to occlude impurities and a small quantity of water.

Although it is generally admitted that N/1 solutions are not sufficiently accurate for analytical work, it is nevertheless the usual practice of acid manufacturers to sell their acids on the acidity of a weighed quantity against N/1 caustic soda, itself standardised against N/1 acid checked against N/1 sodium carbonate.

Accordingly, a N/1 solution of guanidine carbonate (90.07 g. per litre) has been compared with N/1 sodium carbonate and N/1 potassium hydroxide (from mercuric oxide and potassium iodide). The strength of the sulphuric acid was about 0.957 N. The sodium carbonate was Kahlbaum's "pro analysi" dried at 100° C. Solutions were all used at 15° C. in vessels calibrated at that temperature. The following results in g. per l. are the mean of three observations:—

	Sodium carbonate.	Guanidine carbonate.	Potassium hydroxide.
Methyl orange ..	46.853	46.816	46.91
Congo red ..	46.67	46.76	47.1
Alizarin red ..	46.72	46.76	46.78

The acid when standardised against caustic soda itself standardised against N/1 hydrochloric acid (by Gay Lussac's method) with phenolphthalein as indicator gave a result of 46.893 g. per litre. These figures show that the testing of sulphuric acid by means of guanidine carbonate instead of sodium carbonate is accurate enough for routine works practice.

Guanidine carbonate has also been used for the standardisation of N/10 sulphuric acid. Carbon dioxide was not removed, as is advisable for accurate work. The following table gives the factor of approximately N/10 sulphuric acid, each being the mean of three observations:—

Indicator.	Guanidine carbonate.	Sodium carbonate.
Methyl orange ..	1.0075	1.0081
Alizarin red ..	1.0049	1.0075
Congo red ..	1.0060	1.0075

The higher figures with methyl orange are possibly due to a slight acid reaction of the carbon dioxide.

It thus appears that guanidine carbonate should prove a satisfactory and convenient reagent for acidimetry. It merits a more minute investigation, using more delicate indicators, such as bromothymol blue, which are now available.

The Research Laboratory,  
The Alby United Carbide Factories, Ltd.,  
Dagenham.

## THE PREPARATION AND PROPERTIES OF 1.3.5-TRINITROBENZENE.

BY L. G. RADCLIFFE AND A. A. POLLITT.

(This J., 1921, 45 T.)

Dr. W. H. Gibson, of York Street Flax Spinning Co., Belfast, wrote as follows:—"The evidence produced by the authors for the existence of a less stable form of *s*-trinitrobenzene melting at 61° C. fails to convince me. It appears to me that on the occasions when this material was obtained the conditions were such as to favour incomplete nitration, and the low yields recorded support this view. It not infrequently happens that in the trinitration of benzene a mixture of di- and tri-nitrobenzenes is obtained, which is not easily separated by crystallisation, and which gives a product melting in the neighbourhood of 60° C. It seems possible that Messrs. Radcliffe and Pollitt really obtained this mixture, and the behaviour they observed on preparing the aniline and naphthylamine addition compounds is consistent with this, as they do not state that they obtained a quantitative conversion of the unstable to the stable form through the addition compound, nor do they state that they got equal yields of addition compound from equal weights of the two forms. It may be, then, that only a separation of the 1.3.5-trinitrobenzene in a mixture of di- and tri-nitrobenzenes was effected. The analysis of the unstable form by the Knecht and Hibbert method is stated to correspond with pure trinitrobenzene, but it seems unfortunate that this was not confirmed by a nitrogen determination carried out in the usual manner. Quantitative conversion of one form into the other would, however, be the most convincing proof of the existence of this unstable form.

Mr. RADCLIFFE replied as follows:—"I fear that in condensing the paper, certain details have been left out, but we made many experiments involving fractional crystallisation of the product melting at 61° using different solvents, but entirely failed to resolve it or indeed change its melting point; further, we thought at the time that it might be a mixture, and the experiments with aniline etc. and regeneration were done most carefully and with due regard to the quantitative recovery of the trinitrobenzene. I find, from the original notes, that we used several different methods for the determination of nitrogen, and these gave results pointing to the compound being a trinitrobenzene.



## Yorkshire Section.

Meeting held at Leeds on November 8, 1920.

DR. L. L. LLOYD IN THE CHAIR.

### OCBRE STREAMS OF THE VALLEYS OF THE DON AND LOXLEY.

BY J. HAWORTH AND J. EVANS.

Ochre waters may be defined as natural waters in which salts of iron occur in solution. In most cases such waters deposit iron oxide or ochre on the banks and bed of the stream. They may be conveniently sub-divided as:—(1) Chalybeate waters, which occur either as waters containing carbon dioxide and iron bicarbonate, together with other salts, or as saline acidulous chalybeates containing chiefly sodium sulphate and iron bicarbonate. (2) Waters containing chiefly sulphates of iron.

The chalybeate waters seldom contain large quantities of iron (8 to 15 parts per 100,000). They emanate from strata containing iron in the form of carbonate, which in the presence of carbon dioxide forms the soluble bicarbonate. It is not, however, proposed to deal further with this class of waters, but rather with the second class, which are common in the neighbourhood of Sheffield.

Waters containing sulphates of iron are frequently found in all districts where coal workings exist or where outcrops of the coal measures occur. The streams in valleys draining areas in which coal measures are exposed frequently bear marked indications of the presence of iron salts by the brown and red deposits of ochre on the beds of the streams and by the brown colour of the water.

During the course of certain investigations respecting the water supplies of the city of Sheffield the authors made extensive examinations of the contributory streams to the rivers Loxley and Don. The river Loxley is a tributary of the Don, and the watersheds are divided by a precipitous ridge in which outcrops of the lower coal measures occur at numerous points and are exposed. Beneath the lower coal measures beds of ganister and fireclay occur. These are worked at numerous points on both sides of the ridge, the products being largely used in the Sheffield industries. The mining operations consist usually of driving levels or adits into sides of the valleys at different levels where the outcrops occur, and water drains from these forming streams which flow down to the river. There are also other streams flowing from the slopes of the valley, but a casual observation shows that the streams from the mines or ganister pits all contain salts of iron and deposited ochre, whereas the remaining streams contain no iron.

The physical appearance of the ochre streams is very striking. In some cases the water is clear but coloured, the colour varying from pale yellow to a deep reddish-brown; in other cases the water is turbid through the presence of iron hydroxide, but in all cases heavy deposits of iron hydroxide or basic iron sulphate are present.

Between two points in the Loxley Valley about two miles apart, some twelve streams were carefully examined. The following analytical figures (see table) are typical for the water from one of the streams.

The acidity in terms of sulphuric acid varies from 5.57 to 155.57 parts per 100,000, and the total solids in solution from 47.0 to 521.0 parts per 100,000. It will be seen from the analyses that the water

initially contains iron chiefly as ferrous sulphate, which at the point of discharge to the river has undergone practically complete oxidation.

	Parts per 100,000.		
	(1)	(2)	(3)
Total solids .. ..	260.7	410.0	521.6
Solids in suspension:			
Total .. ..	5.7	NIL	NIL
Mineral .. ..	3.5	—	—
Solids in solution:			
Total .. ..	255.0	410.0	521.6
Mineral .. ..	163.0	372.0	480.3
Acidity (as $H_2SO_4$ ) ..	49.0	73.4	112.7
Iron, total .. ..	23.1	13.4	53.0
.. ferric .. ..	20.3	10.2	33.0
.. ferrous .. ..	2.8	3.2	20.0
Hardness (as $CaCO_3$ ) ..	146.8	200.3	—

	Parts per 100,000.	
	Ochre water at entrance to ganister pit, Bower Plantation.	Ochre water taken at point of entry into river.
Total solids .. ..	64.0	506.0
$FeO$ .. ..	8.6	63.2
$Fe_2O_3$ .. ..	22.4	64.9
$CaO$ .. ..	15.0	19.0
$MgO$ .. ..	17.3	14.4
$SO_4$ .. ..	125.7	223.6

The source of the iron salts is without doubt ferric sulphide (iron pyrites) between the layers of the coal strata. Specimens of the shale and coal when first exposed show distinct layers of pyrites in the course of oxidation; in splitting the layers perfect crystals of transparent calcium sulphate in the form of selenite,  $CaSO_4 \cdot 2H_2O$ , may be observed. This substance also finds its way into the water, as the analyses indicate.

The waters issuing from the mines contain ferrous and ferric sulphates and free sulphuric acid, the primary oxidation products of pyrites. In the streams further reactions occur producing the heavy red deposits which consist of ferric oxide and basic ferric sulphates. In some cases the banks consist of this deposited ochre for several inches in depth.

The ochre deposits have their origin in several ways: (1) Direct oxidation and the production of ferric hydroxide; (2) alkaline surface waters joining the stream; (3) the action of iron bacteria, which grow profusely in such waters.

The first two methods of deposition are purely chemical; the third is of considerable interest. Water containing iron salts, even in very minute quantities, forms a very suitable medium for the growth of the organisms commonly referred to as "iron bacteria": two common varieties of these are *Crenothrix polyspora* and *Leptothrix ochracea*. As little as one part of iron in 2 to 3 millions of water is sufficient to permit the development of these organisms. The organisms decompose the salts of iron, depositing the iron in the form of ferric oxide, the liberated acid then attacking the iron of the water pipes. Serious corrosion may occur in this manner.

In the case of ochre streams such as those under consideration the organisms deposit the ferric oxide and the streams become acid. The organisms have very high chemical energy and are able to oxidise ferrous salts many times the weight of their own cells. They are remarkable by reason of the fact that their existence is maintained largely by inorganic salts and the presence of only minute quantities of organic matter is necessary. In the case of the two rivers mentioned the usual flora and fauna are entirely absent.

It will be apparent that the industrial users of waters of rivers into which ochre streams discharge must be subject to great inconvenience and expense. Such waters must be neutralised and

softened before use, or serious corrosion of boilers, pipes, tanks, and iron and steel work will generally occur.

In the case of the rivers under consideration a pure stream of water is within two miles rendered practically unsuitable for industrial use of any kind, except after treatment. Such discharges cannot in any way be controlled or purification insisted upon, as waters draining from mines are specifically excluded from control of rivers authorities under the various Rivers Pollution Prevention Acts.

#### DISCUSSION.

Mr. W. McD. MACKAY mentioned the case of a water supply containing traces of iron as bicarbonate, which after flowing about 100 yards in a shallow stream, became free from iron.

Mr. C. P. FINN gave an instance of an ochre water derived from a disused coal pit, which, unlike the waters tested by the authors, was invariably alkaline. It was quite clear when first pumped, but immediately commenced to precipitate ochre when turned into the stream. In twelve months about 100 yards of pump main was reduced in effective diameter from 10 in. to 2 in. by the deposit.

The CHAIRMAN instanced a water supply at Blackburn which contained a small amount (some 0.3 part per 100,000) of iron, which was sufficient to cause "iron spots" on dyed fabric. Its elimination was attended by considerable difficulty, attributed to the presence of the tannin substances. It had finally been found necessary to harden the water, soften it, and then treat with permanganate.

Dr. CHAPLIN said that in his experience with Wakefield water organic matter high in nitrogen delayed the softening process, and the organic matter tended to keep iron in solution.

Mr. EVANS, in reply to a question, said that the determination of acidity in the presence of iron was difficult, and that so far the authors had found no method that was entirely satisfactory. With reference to bacterial deposits in water mains, he recalled the trouble experienced in conveying water from Lake Vyrny to Liverpool, which had been overcome by efficient filtration at the lake.

Mr. HAIGH JOHNSON said he had found that the bacterial growths in cast-iron water mains were generally confined to places where manganese was present, and he concluded that manganese was essential for the development of these bacteria.

Mr. A. R. TANKARD suggested that the acidity in presence of iron might be estimated by titration with standard lime-water, using phenolphthalein as indicator.

Dr. LLOYD thought that it might be better to replace the phenolphthalein by Congo red dyed a pale shade on silk.

Mr. LOWSON mentioned the hydrogen sulphide method for estimating free acid in the presence of iron.

Mr. HAWORTH said that manganese was always present in small quantities in the waters they had examined.

## Communications.

### SOME METHODS OF PURIFICATION OF *o*-TOLUENESULPHONAMIDE.

BY PHYLLIS VIOLET MCKIE.

In the preparation of saccharin from toluene two nearly related routes (Fahlberg, G.P. 224,386, *et seq.*; and Lange, G.P. 57,391 *et seq.*) may be fol-

lowed. In the Fahlberg procedure the initial stage gives rise to a mixture of *o*- and *p*-sulphochlorides in the ratio of 60:40, while in the alternative Lange process a mixture of *o*- and *p*-sulphonic acids in the same ratio is obtained. The essential difference is that separation of the *ortho* and *para* derivatives is performed in the latter process at the sulphonic acid stage, and in the Fahlberg process partly at the chloride, partly at the amide stage. By partial separation of the chlorides a mixture containing 70% *o*- and 30% *p*-sulphonic chloride is obtained, and these proportions are maintained during the subsequent amidation.

Several methods have been devised by which the proportion of *para* component in this 70/30 *o-p* amide mixture can be reduced:—(1) Precipitation by acids from alkaline solution (G.P. 78,881, 1892). (2) Separation based on the fact that of the crystalline salts formed with alkalis, that of the *ortho* compound is the less soluble (G.P. 77,435, 1894). (3) Precipitation from alkaline solution by ammonium salts (G.P. 154,655, 1903). A fourth process, which involves the transformation of the amides again into the sulphonic chlorides, has also been used (G.P. 133,919, 1902).

In no case do these patents claim complete separation. A certain amount of *o*-toluenesulphonamide, still mixed with 7–10% of *para* component, and hence of only 90–93% purity, is extracted, while the residual mother liquor contains, besides the bulk of the *p*-toluenesulphonamide, a varying proportion—up to 30%—of the *ortho* isomer. Oxidation of the mixture containing only 90% *ortho* compound thus produces a saccharin of equal impurity.

On the other hand, separation of the *ortho*- and *para*-toluenesulphonic acids in the Lange method of preparing saccharin produces an acid containing already 90% of *ortho* compound. Subsequent chlorination and amidation of this acid thus yields a crude *o*-toluenesulphonamide mixed with only 10% *para* compound.

It was the object of this investigation to examine and test a number of simple methods of freeing the *o*-toluenesulphonamide from this 10% of *para* derivative, which remains after the preliminary separation of the 70/30 mixture by the patented processes. Especial emphasis is laid on the avoidance of loss of *o*-toluenesulphonamide in the purification.

#### Crystallisation from water.

(The solubility in water of *o*-toluenesulphonamide is 1 part in 958 parts at 9° C.; *p*-toluenesulphonamide, 1 part in 515 parts at 9° C. Klason, Vallin. Ber., 1879, 12, 1851.)

One recrystallisation of the crude (90% pure) amide mixture from water sufficient to retain the whole of the contained *p*-toluenesulphonamide in solution yields 80% of *o*-toluenesulphonamide of 97–98% purity.

TABLE I.

Initial mixture.	Recrystallised material.	m.p.	Purity.
5 g.		150°	90%
1st	4 g.	155°	98%
2nd	3.6 g.	155.2°	98.3%
3rd	3.06 g.	155.5°	99.2%

Only by fractional sublimation *in vacuo* could the melting point be raised above that obtained by a third recrystallisation from water. (The melting point of pure *o*-toluenesulphonamide is 156.3° C. McKie. Chem. Soc. Trans., 1918, 113, 799.)

*Precipitation by acids from alkaline solution.*

In considering the separation of *o*- and *p*-toluenesulphonamides from alkaline solution, or the extraction of the solid amides by aqueous alkali, two opposing factors have to be taken into account. Of the two amides the *ortho* compound is the less soluble, as is also the sodium salt of the *ortho* amide, but *o*-toluenesulphonamide should, by virtue of its configuration, possess the greater "acidity" (*cf.* *o*-chlorobenzoic and *o*-nitrobenzoic acids, which are much stronger acids than the corresponding *para* derivatives).

The solid toluenesulphonamide mixture, containing 90% *ortho* and 10% *para* derivative, was dissolved in a solution of one mole of 8–10% sodium hydroxide. Addition of acids of varying strengths and in varying proportions precipitated corresponding amounts of solid toluenesulphonamide, which was collected, dried, and weighed in a Gooch or alundum crucible. As there is always a slight loss in the steam oven, the solid was dried over sulphuric acid *in vacuo* at 50° C.

(i) *Acid equivalent to one-tenth of o-toluenesulphonamide*.—Addition of acid equivalent to one-tenth of the *o*-toluenesulphonamide brings out almost completely pure *ortho* compound, largely independent of the hydron concentration of the acid. Except for very low concentrations of hydron, solubility is the most powerful factor, and hence the less soluble—the *o*-toluenesulphonamide—is precipitated only very slightly contaminated with *para* derivative. But with such a slightly ionised acid as carbonic acid the relative acidities of the two toluenesulphonamides come into play, though solubility is still dominant, hence a small amount (3% by carbonic acid) of *p*-toluenesulphonamide is precipitated.

TABLE II.

5 g. of mixed toluenesulphonamide, containing 10% *p*-toluenesulphonamide, in 1 mole (1.17 g.) NaOH in 15 c.c. of water.  
Acid equivalent to *p*-toluenesulphonamide added in 10 c.c. of water.

Acid.	Hydrochloric	Acetic.	Carbonic.
Wt. amide precipitated ..	0.53 g.	0.5263 g.	0.4820 g.
Melting point ..	156°	155°	154.5°
% <i>o</i> -toluenesulphonamide in precipitate ..	99.5	98	97

(ii) Experiments were made in which amounts of hydrochloric and acetic acid equivalent to 70% of *o*-toluenesulphonamide were added to solutions of mixed amide containing 10% of *para* compound, in one mole of 8–10% sodium hydroxide. After standing for some hours the precipitated solid was filtered off. A second and third separation was obtained by addition of acid respectively equivalent to 15% and 9% of *o*-toluenesulphonamide.

TABLE III.

	Hydrochloric acid.	Acetic acid.
Wt. mixed amide, m.p. 150° ..	5 g.	5 g.
Acid=70% of <i>o</i> -amide ..	8.2 g.	3.158 g.
Amide precipitated ..	154°=(98% <i>o</i> -amide)	156°=(99.5% <i>o</i> -amide)
Melting point ..	154°	156°
Acid=15% of <i>o</i> -amide ..	0.65 g.	0.65 g.
Amide precipitated ..	150°=(90% <i>o</i> -amide)	155°=(98% <i>o</i> -amide)
Melting point ..	150°	155°
Acid=9% <i>o</i> -amide ..	—	0.489 g.
Amide precipitated ..	—	149°=(87.5% <i>o</i> -amide)
Melting point ..	—	149°

As will be seen from the table, the weaker acid, acetic, gave a precipitation of *o*-toluenesulphon-

amide, containing only 0.5% of *para* component, and representing 65% of the total amide originally used. By addition of acetic acid equivalent to a further 15% of *o*-toluenesulphonamide a solid still containing 99% of *ortho* component was obtained.

On the other hand, the strong acid, hydrochloric acid, gave in the first separation a solid containing 3% of *p*-toluenesulphonamide, and in the second (using acid equivalent to 15% *o*-toluenesulphonamide), a mixture containing 10% of *para* derivative. Hence it is obvious that too high a concentration of hydron so puts back the ionisation of each of the amides that their relative acidities cease to be operative, and the amides are precipitated in a proportion dependent solely on their relative solubilities.

*Extraction of the solid mixture by alkali.*

The solid toluenesulphonamide mixture was ground up with the requisite quantity of aqueous alkali at a temperature of 50° C., and shaken at that temperature at intervals for three hours. It was then left at room temperature for sixteen hours, after which it was filtered.

A comparison on these lines between sodium hydroxide and sodium carbonate showed conclusively that the weaker alkali, sodium carbonate, effects a cleaner separation than does the stronger sodium hydroxide, and the recovery of pure *o*-toluenesulphonamide is therefore higher.

TABLE IV.

10 g. mixed toluenesulphonamides, containing 10% *p*-toluenesulphonamide. m.p. 150°.

	NaOH.	Na <sub>2</sub> CO <sub>3</sub> .
Alkali (conc. 1.17%) = <i>p</i> -amide ..	0.234 g.	0.308 g.
Amide undissolved ..	8.74 g.	9.374 g.
Melting point ..	152.5°	153.5°
% <i>o</i> -amide in undissolved amide ..	93.5	95

If the concentration of the alkali be increased, relatively more *p*-toluenesulphonamide is extracted, and hence the undissolved amide contains a higher proportion of *ortho* compound. But the loss (dissolved material) increases with increase of concentration, so that the higher the concentration of the alkali, the less is the total remaining amide (*v.* Table V). While at a concentration of 7.7% alkali, extraction with two moles of sodium carbonate yields a slightly purer *o*-toluenesulphonamide than does a concentration of 2.3%, nevertheless the percentage of dissolved solid is double that at the lower concentration.

TABLE V.

*Effect of concentration.*

Mixed amide taken, 10 g. (90% *o*-amide). m.p. 150°.

Concentration of Na <sub>2</sub> CO <sub>3</sub> .	1.17%.	2.3%.	7.7%.
Undissolved amide ..	9.38 g.	9.28 g.	9.02 g.
Melting point ..	153°	153.5°	154°
<i>o</i> -amide in undissolved solid ..	94%	95%	96%
Amide from mother liquor by acidification ..	0.3 g.	0.3 g.	0.29 g.
Melting point ..	115°	109°	117°
<i>p</i> -amide present ..	69%	60%	68%
Loss ..	3%	4%	6%

*Extraction of the solid mixture with aqueous alcohol.*

(1 part *o*-toluenesulphonamide dissolves in 28 parts of alcohol, 1 part *p*-toluenesulphonamide dissolves in 13.5 parts of alcohol at 5° C. Klason, Vallin. *Ibid.*)

The mixed toluenesulphonamides were ground up with 96% alcohol, sufficient to dissolve the whole of the contained *p*-toluenesulphonamide. A residual solid representing 83·8% of the total material, but containing only 95% of *o*-toluenesulphonamide, was thus obtained.

The use of 50% aqueous alcohol is more successful. The undissolved amide, which represents 93·5% of the *o*-toluenesulphonamide, in the original mixture, consists of 98% of *o*-toluenesulphonamide and 2% of *p*-toluenesulphonamide.

TABLE VI.

10 g. mixed toluenesulphonamide, containing 1 g. *p*-toluenesulphonamide.

	95% alcohol.	50% alcohol.
Amide undissolved .. ..	8·38 g.	8·403 g.
Melting point .. ..	153·5°	155°
<i>o</i> -amide in undissolved amide	95%	98%

#### Purification by partial oxidation.

In that process of oxidation of *o*-toluenesulphonamide to saccharin in which *o*-toluenesulphonamide in excess of potassium permanganate is used (E.P. 3563, 1903), the excess amide is isolated as completely pure *o*-toluenesulphonamide. Any *p*-toluenesulphonamide present is oxidised to *p*-sulphaminobenzoic acid. Application of this procedure as a means of purification of a 90% mixture of *o*- and *p*-toluenesulphonamides was highly successful. 90–94% of the *o*-toluenesulphonamide was obtained in a high degree of purity. In the process saccharin of a like degree of purity, equivalent to 5–9% of *o*-toluenesulphonamide, is also isolated. Hence the total loss—unrecovered material—is only the 10% represented by the initial *para* component.

Economically, however, the loss of permanganate necessitated by this procedure is an important factor. The saccharin isolated represents only 60% of the potassium permanganate reduced.

TABLE VII.

10 g. mixed toluenesulphonamide, containing 1 g. <i>p</i> -toluenesulphonamide.	
Potassium permanganate equivalent to 1 g. <i>p</i> -amide=1·84 g.	
Unoxidised amide .. ..	= 8·1 g. m.p. 155·5°=99·2% <i>o</i> -amide
Saccharin .. ..	= 0·93 g. m.p. 226°=0·87 g. ..
Loss—i.e., unrecovered material .. ..	= 1·03 g.

The *o*-amide is represented by 99·2% of 8·1 g. unoxidised=8·0595 g. and saccharin equivalent to 0·87 g., or a total of 8·9295 g. Hence the loss of *o*-toluenesulphonamide is 0·07 g.

#### Conclusions.

Consideration of the results of the various methods of separation of *o*- and *p*-toluenesulphonamides shows that certain processes stand out as yielding a product of greater purity and with a minimum loss.

In every case final purification, brought about by repeated recrystallisation from water, followed by sublimation *in vacuo* is required.

The highest degree of purity which can be obtained is effected by precipitation by acetic acid of 70% of the *o*-toluenesulphonamide from an alkaline solution of the mixed amides; a product containing 99·5% of *o*-toluenesulphonamide is thereby obtained. But the loss involved is very high—30% of the *ortho* compound.

Purification by partial oxidation gives, on the whole, the most satisfactory results. A product of a high degree of purity—99%—is obtained, and including the saccharin which is isolated, represents the whole of the valuable *o*-toluenesulphonamide,

a fact of immense importance in the manufacture of saccharin. But the many and lengthy operations involved, and the fact that for every one part of *o*-toluenesulphonamide recovered 0·216 part of potassium permanganate is destroyed, and of this 40%, or 0·086 part, is not accounted for by any product isolated, somewhat detracts from the advantages which this method possesses.

For simplicity and rapidity of manipulation, combined with a good yield of *o*-toluenesulphonamide of a high degree of purity, one extraction of the mixture with 50% aqueous alcohol (1 part of mixed amide to 5 parts of 50% alcohol) is to be recommended. By this means 90·7% of the *o*-toluenesulphonamide of 97% purity is extracted.

Extraction of the mixture with a moderately concentrated solution of sodium carbonate, while it does not effect such a clean separation (the *o*-toluenesulphonamide is of only 96% purity), brings out almost the whole of the *ortho* compound; a loss of but 2–3% is involved.

On these grounds, together with its low cost and ease of manipulation, it may be suggested as an admirable means of separating mixtures containing more than 10% *para* compound, and even of the "4–3" mixture obtained in the Fahlberg saccharin process.

I desire to express my indebtedness to Professor Orton, under whose direction this work has been carried out.

University College of North Wales,  
Bangor.

#### THE VOLATILE OIL FROM THE LEAVES OF THE "WILD PIMENTO" OF JAMAICA.

BY O. D. ROBERTS, F.I.C.

The genus *Pimenta* includes two species which are well known in commerce as sources of volatile oils. These are (1) *P. officinalis*, the unripe fruit of which yields ordinary pimento or allspice oil, whilst the leaves also furnish an oil of similar composition; and (2) *P. acris*, from the leaves of which commercial oil of bay is obtained.

Two volatile oils from the leaves of sub-varieties of *P. acris* have also been examined, viz., oil of *P. acris* var. *citrifolia*, Kostel, which is said to contain an appreciable quantity of citral, and oil from a form of *P. acris*, known as "Bois d'Inde anise," which has a characteristic anise-like odour.

The oil which is the subject of the present investigation differs widely in composition from any of the above oils, and has an odour recalling that of spike lavender oil. It is derived from the leaves of a species of pimento found in Jamaica, both in the woods of Upper Clarendon at an altitude of 2000–2500 feet, and in the Dry Harbour Mountains. The tree attains a height of 40 feet and its trunk a diameter of 1 foot. The species is locally known as "Wild pimento," and has been named *Amomis* (= *Pimenta*) *jamaicensis*, Britton and Harris, by the Government botanist.

Leaves distilled in Jamaica were stated to have yielded 0·49% of oil. A sample of the oil received at the Imperial Institute from the Director of Agriculture of Jamaica in 1918 was pale yellow, and had an odour somewhat resembling that of the oil of *Lavandula spica*. It had the following constants: sp. gr. (15°/15° C.) 0·8895;  $n_D^{20}$  (in a 100 mm. tube) = –6° 0';  $n_D^{25}$  = 1·471; acid value 2·4; ester value before acetylation 4·2; ester value after acetylation 129·4. The oil was soluble in 2½ volumes of 70% alcohol at 15° C., becoming cloudy with 6 volumes.

The following results were obtained in a systematic investigation of the oil.

*Free acids: Indication of the presence of caproic and acetic acids.*

The free acids were extracted from the oil by shaking it with a dilute solution of sodium carbonate. On acidifying the solution and extracting with ether, an amount equivalent to about 0.2% of the original oil was obtained. This acid liquid was distilled in a current of steam, when there separated in the distillate a small quantity of an acid substance having an unpleasant odour resembling that of caproic acid. After separation from the aqueous portion of the distillate, this substance was converted into its silver salt, of which 0.1270 g. gave 0.0617 g. Ag (Ag=48.6%). Silver caproate contains 48.4% Ag. The aqueous portion of the distillate was saturated with calcium chloride, washed with ether, and again distilled in a current of steam. The distillate contained a readily soluble acid; 0.1023% of its silver salt gave 0.0657 g. Ag (Ag=64.2%). Silver acetate contains 64.6% Ag.

*Aldehydes.*

On shaking the oil with a saturated solution of sodium hydrogen sulphite, combination to the extent of about 0.1% of the original oil took place. The substance thus removed from the oil had a rather pleasant odour, and was aldehydic in character, for it responded to Schiff's rosaniline test. The amount of liquid obtained was not sufficient for further investigation.

*Phenols. Absence of eugenol.*

The original oil, after the foregoing treatment with sodium carbonate and sodium hydrogen sulphite solutions, was extracted with a 3% solution of sodium hydroxide. A phenolic liquid was obtained which amounted to about 0.1% of the original oil. This substance had a slight phenolic odour, and produced a reddish-violet coloration with a solution of ferric chloride, but no evidence of the presence of eugenol was obtained.

*Fractional distillation of the oil.*

Before proceeding with the fractional distillation of the oil remaining after the removal of the above-mentioned traces of free acids, phenols, and aldehydes, the oil was hydrolysed by boiling it with alcoholic potassium hydroxide. After the greater portion of the alcohol had been removed by distillation, sufficient water was added to effect the separation of the oil, which was extracted with ether. The ethereal solution was washed and dried, the ether removed, and the oil submitted to repeated fractionation. The following fractions were obtained:—

	Per cent. calculated on the original oil.	Range of boiling point.		Sp. gr. (15°/15° C.)	n <sub>D</sub> <sup>20</sup> 100mm. tube.	n <sub>D</sub> <sup>25</sup>
		Under 14 mm. pressure.	Under atm. pressure.			
1.	32	75°–80°	175°–190°	0.8750	–8.25°	1.4720
2.	42	80°–95°	190°–205°	0.8677	–3.74°	1.4670
3.	4	95°–110°	205°–220°	0.8808	–2.30°	1.4795
4.	6	110°–125°	220°–235°	0.9010	–3.80°	1.4865
Residue 16	—	—	—	0.9592	Too dark to obtain a reading.	1.5205

*Identification of cineol.*

The fraction boiling at 175°–190° C. was a colourless limpid liquid, having a camphoraceous odour. On treating it with a 50% solution of

resorcinol, an absorption of 47% was obtained, equivalent to 15% of the original oil. The remaining oil was separated from the clear resorcinol solution, and the latter was then subjected to distillation with steam. The colourless oil, which separated in the distillate, had the characteristic odour of cineol, was optically inactive, had sp. gr. (15°/15°) 0.9270, and furnished a pale greenish-white iodole addition compound, which melted and decomposed at 116° C. It was evident therefore that the portion of this fraction absorbed by the resorcinol solution consisted practically entirely of cineol.

*Identification of  $\alpha$ -phellandrene.*

The part of this fraction (175°–190°) which was unabsorbed by the resorcinol solution was distilled over sodium under reduced pressure, and a colourless mobile oil was thus obtained which had sp. gr. (15°/15°) 0.8520,  $n_D^{25}$  (in a 100 mm. tube) = –11° 42'; b.p. 172°–179° (uncorr.). A portion of this oil, treated with sodium nitrite and glacial acetic acid, gave the characteristic reaction of phellandrene. The nitrite, after crystallisation from methyl alcohol and chloroform, melted at 112°–113° C., thus establishing the presence of  $\alpha$ -phellandrene in the oil.

*Identification of dipentene.*

From another portion of the above oil, boiling at 172°–179°, a fraction boiling at 176°–179° was obtained, which furnished a tetrabromide melting at 124° C., thus proving the presence of dipentene.

*Identification of linalool.*

The fraction boiling at 190°–205°, which was the largest fraction obtained, consisted of a colourless liquid, with a pronounced odour of linalool. Almost the entire fraction boiled at 197°–198° C.; this portion had sp. gr. (15°/15°) 0.8693,  $n_D^{25}$  = –3° 39',  $n_D^{20}$  = 1.4655, which are the characteristic physical constants of linalool. A portion was gently oxidised with chromic acid mixture, and the resulting oil, after purification by distillation with steam, was treated with sodium hydrogen sulphite solution. From the solid bisulphite compound thus produced, citral was liberated, which was recognised by its odour, and identified by the preparation of crystalline  $\alpha$ -citryl- $\beta$ -naphthacinchoninic acid, m.p. 200° C. From these results it is evident that this fraction consisted almost entirely of linalool.

The small fraction boiling at 205°–220° C. was a colourless liquid, possessing a pleasant aromatic odour. The alcoholic nature of this fraction was evidenced by its almost complete esterification when treated with acetic anhydride; the combination which took place on heating a portion with phthalic anhydride was, however, almost negligible, thus indicating that the substance consisted of a tertiary alcohol. It was clear that this fraction also consisted largely of linalool, and no definite indication of the presence of any other alcohol was obtained.

*Identification of geraniol.*

The fraction boiling at 220°–235° consisted of a colourless liquid, possessing a faint rose-like odour. When a few drops of this fraction were oxidised with chromic acid mixture the strong odour of citral was developed. The fraction, diluted with an equal volume of benzene, was heated for several hours with phthalic anhydride. The acid phthalic ester thus formed was converted into the sodium salt which, on saponification and simultaneous distillation in a current of steam, furnished an oil having the characteristic odour of geraniol. The presence of geraniol was confirmed by the formation of geraniol diphenylurethane, m.p. 80°–81° C.

The fraction boiling above 235° was brown and rather viscous. It was evident that it contained resinous decomposition products, consequent upon distillation of the oil under atmospheric pressure. With the object of ascertaining whether any characteristic sesquiterpene was present, this fraction was heated on the water bath for some weeks with sodium under reduced pressure. The oil was then distilled off under 15 mm. pressure. There was thus obtained a colourless oil, with a slight balsamic odour, having sp. gr. (15°/15°) 0.9320, and boiling between 245° and 290° (uncorr.) at atmospheric pressure. Its high density, wide range of boiling point, and the fact that the oil was still slowly attacked by sodium, showed that appreciable quantities of oxygenated compounds were still present. The amount of oil was too small to permit of any further investigation of this fraction.

#### Summary.

The results of this investigation of the volatile oil from the leaves of *Aromis jamaicensis* have shown that its composition is approximately as follows:—Terpenes, including  $\alpha$ -phellandrene and dipentene, 17.0%; cineol, 15.0%; aldehydes, undetermined, 0.1%; alcohols, chiefly l-linalool with some geraniol, 38.3%; esters, as linalyl acetate, 1.5%; phenols, undetermined, 0.1%; acids (acetic and caproic in the free state), 0.2%; residue probably containing sesquiterpenes, 27.8%.

Scientific and Technical Research Department,  
Imperial Institute.

#### NOTE ON A SPECIAL SLIDE RULE FOR CALCULATION OF "WET" AND "DRY" WEIGHTS OF DAMP MATERIAL.

BY R. SCOTT.

The necessity for a simple, rapid, and accurate method of calculating the "wet" and "dry" weights of nitrocellulose latches led to the construction in this laboratory of a calculator which has proved useful, and the principle employed seems to offer a wider scope for practice.

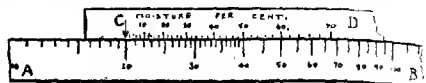
A slide rule may be used in many cases where a nomographic or alignment chart has been suggested for routine calculations. It is easily constructed and easily manipulated, and the time required for the calculation is reduced to a few seconds.

In handling a batch of damp material it is customary to have a sample submitted for determination of moisture which is expressed as moisture per cent. of the damp material. There is now required the amount of "wet" material to give a specific amount of "dry" or the "dry" content of a certain amount of "wet."

The calculation is given by

$$D \times \frac{100}{100-M} = W$$

where D=dry weight, W=wet weight, and M=moisture per cent. This is of the general form  $XY=Z$  and capable of slide rule or nomographic treatment.



To make the slide rule the frame scale, A B (see fig.), was divided in the usual logarithmic manner but the slide scale, C D, was divided proportionally to  $\log. 100/(100-M)$  between suitable limits of M, each division being marked with its appropriate value of M. The frame scale, A B, was extended beyond the point marked 100 by a length equal to the total length of the slide scale.

For wet weight determination the zero mark on C D is placed opposite the division on A B corresponding to the "dry" weight, and the "wet" weight read off opposite the division on C D marked with the moisture content. For "dry" weight determination the process is reversed.

A convenient distance between the points marked 10 and 100 on the frame was found to be 1000 mm.

The wooden frame and slide were constructed in the carpenter's shop and made so that C D could be moved easily along A B. The scales were glued to the wood and coated with celluloid varnish. The whole apparatus measured 4' 9" x 3 1/4" x 3/4".

The accuracy of the instrument, assuming careful construction, will depend on the scale on which it is constructed and the number of divisions introduced. The calculator in use has a total maximum apparent error due to size of scale divisions of 0.56 lb. dry weight on 100 lb. of dry material, but this is due to errors accumulating in the same direction. In practice the reading of figures may be made to approximations, where necessary, between divisions, and the error thus maintained well within the limit.

It is easy to modify the slide scale to read weights where a certain percentage of moisture is allowed, as in air-dry material. The slide in this instance would be divided proportionally to  $\log. (100-a)/(100-M)$ , where  $a$  is the percentage of moisture allowable.

In general a similar instrument should be useful for rapid calculations of the type  $a=x.f(y)$ , with the slide scale divided in proportion to  $\log. f(y)$ .

By using several slides, equations of the type  $\phi(a)=F(x).f(y)$ ... may be solved by dividing the frame scale proportional to  $\log. \phi(a)$  and slide scales proportionally to  $\log. F(x)$  and  $\log. f(y)$ ...

Necol Industrial Colloidions, Ltd.,  
Stowmarket.



## Communications.

## SOME PROPERTIES OF TETRANITRANILINE (TNA).

BY B. J. FLÜRSCHHEIM, PH.D.

Since the appearance of the first publications on TNA<sup>1</sup> many authors or patentees have dealt with the subject. Additional information of value has resulted in some cases, whereas erroneous or incorrect statements have been made in others, and it may therefore be useful to give a brief review of the subject on the basis of seventeen years' personal experience. The present communication will therefore deal with the constitution and reactions of TNA, its density and colour, stability, physiological properties, power, and sensitising properties, and certain points connected with its manufacture. Of the tests recorded below, in all of which, unless otherwise stated, merely water-washed, unrecrystallised TNA manufactured on plant scale has been used, Nos. 2, 4-8, 14-16, 22, 26-29, 42, 43, 49-51, 58-60, and 64 were carried out by Allied and associated Governments, Nos. 9, 21, 25, 30, 35, 56, 57, 62, 63 (partly), by explosives manufacturers, Nos. 44, 52, 55, and 61 by ordnance manufacturers, Nos. 3, 10-13, 17-20, 23, 24, 31-34, 36, and 63 (partly) by the author, and the remainder are quoted from the literature on the subject.

## I.—CONSTITUTION AND REACTIONS OF TNA.

TNA was first prepared and analysed by the author in 1904, and the work was partially repeated by his assistant, T. Simon, in 1906. The determination of the positions occupied by the nitro-groups was carried out by the author in 1910, both by synthesis from derivatives of aniline containing a second nitro-group in an ortho- or para-position with regard to the amino group,<sup>2</sup> and by the conversion of TNA into compounds already known, notably trinitroresorcinol<sup>3</sup> and trinitro-*m*-phenylenediamine.<sup>4</sup> It was thus definitely proved that the nitro-groups occupy the positions 2.3.4.6, that the 3-nitro-group is mobile,<sup>5</sup> also that all are real nitro and not nitrite groups, for otherwise ammonia could not transform TNA to trinitro-*m*-phenylenediamine, but only to an ammonium salt of an aminophenol.

In order to test the nitro-formulation of the mobile group in the 3-position, Van Duin<sup>6</sup> recently reduced TNA by means of titanous chloride, and his results confirmed the already well-established fact. It had already been stated<sup>7</sup> that the mobile nitro-group can be replaced by numerous other radicles—for instance, by reaction with alcohols, phenols, or amines—and that the amino-group can be acetylated, and acetyltetranitraniline, trinitroaminophenol, -anisol, and -phenetol had been described. These compounds were again prepared by Van Duin,<sup>8</sup> as well as some of the amines.<sup>9</sup>

<sup>1</sup> Flürschheim, E.P. 3224 and 3907 (1910); U.S.P. 1,045,011-2; G.P. 243,079 and 241,697; etc., etc. Flürschheim and Simon, Proc. Chem. Soc., 1910, 20, 81. Flürschheim, 8th Int. Cong. Appl. Chem., 1912, Orig. Comm., 4, 31; Z. ges. Schless.-u. Sprengstoffw., 1913, 8, 185.

<sup>2</sup> E.P. 3224/1910.

<sup>3</sup> Proc. Chem. Soc., 1910, 20, 81.

<sup>4</sup> E.P. 3907/1910.

<sup>5</sup> Stettbacher's remarks (Z. ges. Schless.-u. Spreng., 1918, 13, 412), ascribing this proof to Van Duin (Thesis, Utrecht University, 1918; Rec. Trav. Chim., 1919, 38, 89) are therefore incorrect.

<sup>6</sup> Rec. Trav. Chim., 1920, 39, 578.

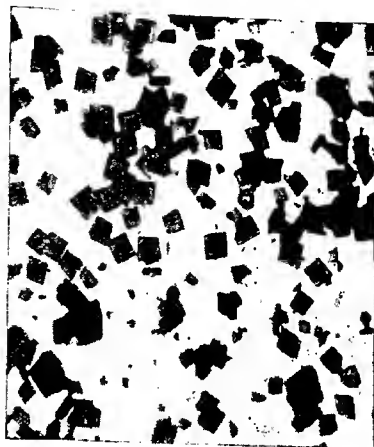
<sup>7</sup> Proc. Chem. Soc., 1910, 20, 81. Z. ges. Schless.-u. Spreng., 1913, 8, 185. E.P. 3907/1910; Ber., 1914, 47, 426.

<sup>8</sup> Rec. Trav. Chim., 1920, 39, 148, with Van Lennep. These authors overlook the prior publications concerning trinitroaminophenol.

<sup>9</sup> Rec. Trav. Chim., 1919, 38, 89. Van Duin's melting points are corrected for the exposed stem, whereas those previously given by the author are the usual uncorrected ones.

## II.—DENSITY AND COLOUR.

The true colour of TNA is a strong yellow, which always appears in the ground material. Larger crystals, however, exhibit a deeper hue, varying, according to the mode of preparation, from an intense yellow to greenish. The micrograph obtained by Col. C. G. Storm, at the U.S. Bureau of Mines.



at an enlargement of 31 diameters, reveals a flaw in the crystals, and under the microscope the centre of the crystal, with the flaw, appears yellow, the transparent surrounding portion green.

Crystals without a flaw can be obtained under certain conditions of nitration, and these, under the microscope, appear entirely transparent and green. According to the method of nitration the material in bulk, as separated from the spent acid, therefore appears—without a flaw—greenish, and has an absolute density of quite 1.9, or more or less yellow, with a corresponding decrease of the density to anything between 1.9 and 1.86. This observation is chiefly due to Dr. Beardsley, of Bound Brook, N.J.

The absolute density of TNA therefore equals that of ammonium perchlorate (1.89). The absolute densities of all other organic high explosives and of ammonium nitrate lie approximately between 1.6 (nitroglycerin) and 1.77 (trinitrochlorobenzene).

## III.—STABILITY AND REACTIVITY.

As early as 1914 W. Macnab,<sup>10</sup> on the basis of several years' personal experience, was able to state that TNA "combines an exceptional explosive power with aromatic stability." Indeed, no TNA which the author has examined since its discovery in 1904, including even the very first impure samples, has proved unstable in the sense of being unsafe to handle or store. But to produce a material capable of satisfying all the more or less sensitive tests (which were really intended, not for purely aromatic products like TNA or TNT, but for wholly or partly aliphatic nitro-compounds capable of catalytically accelerated spontaneous decomposition, such as nitrocellulose, nitroglycerin, tetryl, etc.) has required the accumulated experience of several years, but has now been achieved on a commercial scale and without any purification of the material other than washing with water.

<sup>10</sup> Lectures on explosives, Institute of Chemistry, pp. 25, 60 (1914).

Typical examples of different TNA stability tests are given below, and the results are subsequently interpreted and compared with those obtained from other nitro-explosives.

#### A.—Abel test.

From each test the approximate percentage of NO<sub>2</sub> which would be evolved per year if the measured initial rate were maintained has been calculated by Robertson and Smart's formula.<sup>11</sup>

No. 1. Carried out by Van Duin and Van Lennep<sup>12</sup> with TNA prepared with KNO<sub>3</sub> and re-crystallised from acetic acid. Used 10 grams in closed containers of 29.3 c.c. capacity, heated without test paper; times given are those after which an inserted starch-zinc iodide test paper is coloured in 30 seconds.

Temp.	Time.	NO <sub>2</sub> % per year. <sup>13</sup>
70° C.	3 hrs.	0.044
60° C.	26 "	0.0051
50° C.	404 " †	0.00038
32° C.	1512 "	0.000087

† 2 samples out of 4.

No. 2. Heated with test paper according to standard American Abel-test method, temperature 65.5° C.—

(a) Standard plant TNA, dried on plant scale 6 hrs. at 80° C. in a partial vacuum of 26–27 in. (4 samples): 4 hrs. 13 min. plus; NO<sub>2</sub> < 0.0007% per annum.

(b) Similar material, original moisture content 0.22%, dried only 1 hr. at 80° C. at ordinary pressure, tested a few days after manufacture: 30, 50, 51 min.; NO<sub>2</sub> av. 0.02% p.a.

(c) Same material as (b), not dried: 17, 26, 41 min.; NO<sub>2</sub> av. 0.05% p.a.

(d) Similar material, not dried, after 9 months' ordinary storage (3 samples): Slight test after 63 min., no increase of tint in further 33 min.; NO<sub>2</sub> 0.009% p.a.

(e) Same material as (a), after adding a drop (ca. 5% of water): 12½, 12½ min.; NO<sub>2</sub> 0.26% p.a.

(f) Same material as (e) after re-drying for 1 hr. at 100° C.: 60 min. plus; NO<sub>2</sub> < 0.01% p.a.

No. 3. Plant TNA, which had been stored 6½ years, with a moisture content of 0.49%, was used and American Government potassium iodide test paper:—

(a) Pre-dried 4 hrs. at 60° C., whereby moisture was reduced to 0.26%. 10 g. TNA, test tube of 39 c.c., temp. 95° C., heated with moist test paper: av. 15 min.; NO<sub>2</sub> 0.05% p.a. at 95° C.

(b) Pre-dried 4 hrs. at 95° C., 10 g. TNA, temp. 95° C., test tube 39 c.c., heated with moist test paper: av. 32 min.; NO<sub>2</sub> 0.011% p.a. at 95° C.

(c) Pre-dried 4 hrs. at 95° C., 2 g. TNA, heated by Van Duin and Van Lennep's method, without test paper, test tube 25 c.c., 24 hrs. at 100° C.: av. 1 min.; NO<sub>2</sub> 0.013% p.a. at 100° C.

(d) Same as (c), 6½ hrs. at 100° C.: av. 3 min.; NO<sub>2</sub> 0.016% p.a. at 100° C.

(e) Same as (c), 6 hrs. at 100° C.: av. 4 min.; NO<sub>2</sub> 0.013% at 100° C.

(f) Same as (c), but only 0.1 g. TNA, 2 hrs. at 130° C.: av. 1½ min.; NO<sub>2</sub> 2.5% p.a. at 130° C.

(g) Same as (f), preheated 14 hrs. at 130° C., heated 2 hrs. at 130° C.: av. 3 min.; NO<sub>2</sub> 1.1% p.a. at 130° C.

#### B.—Manometric and gravimetric heat tests.

No. 4. Gas evolution in *vacuo* of plant TNA after about 10 months' ordinary storage. Pre-dried, before test, for 4 hrs. at 60° C., 18 hrs. at 80° C.: 1st hour, 0.60 c.c. per g. per hr. at 120° C.; 2nd,

0.25; 3rd, 0.16; 4th to 7th, 0.11; 8th to 11th, 0.09; 12th to 15th, 0.07; 16th to 19th, 0.06; 20th to 30th hour, 0.052 c.c.

No. 5. Same material after about 45 months' storage. Pre-dried 18 hrs. at 30°–40° C., 2 hrs. at about 90° C.: 1st hour, 0.65 c.c. per g. per hr. at 120° C.; 2nd 0.28; 3rd, 0.24; 4th to 7th, 0.16; 8th to 11th, 0.10; 12th to 15th, 0.075; 16th to 19th, 0.06; 20th to 30th hour, 0.044 c.c.

No. 6. The same material as No. 5, after grinding and water-washing, then pre-drying as in No. 5, gave results practically identical with those of No. 5.

No. 7. Same material as No. 4, similarly pre-dried: 1st 16 hrs., 0.034 c.c. per g. per hr. at 100° C.; 17th to 32nd, 0.020; 33rd to 48th, 0.015; 49th to 64th, 0.0125; 65th to 80th, 0.0112; 81st to 96th, 0.0106 c.c.

No. 8. TNA from an experimental run on a small plant scale, in which a mixed acid had been used containing about 2% more water than specified for standard TNA. Pre-dried 18 hrs. at 30°–40° C. and 2 hrs. at 90°–100° C.: first ½ hr., 8.8 c.c. per g. per hr. at 120° C.; ½ hr.—3 hrs., 1.28; 4th to 19th, 0.25; 20th to 27th hrs., 0.15 c.c.

No. 9. TNA made on an experimental scale<sup>14</sup>: 1st 4 hrs., 0.11 c.c. per g. per hr. at 120° C.; 5th to 8th, 0.075; 9th to 12th, 0.045; 13th to 16th, 0.035; 17th to 20th hrs., 0.030 c.c.

No. 10. The loss of weight of TNA (same material as No. 4, pre-heated 3 hrs. at 96° C.) was found to be as follows:—At 130.5° C., first 2 hrs., 0.113% per hr.; 2 hrs. to 9h. 40m., 0.031%; 9h. 40m. to 17h. 20m., 0.017%; 17h. 20m. to 25 hrs., 0.012%. Thereafter at 105.5° C., first 5½ hrs., 0.0012%; 5½ to 10½ hrs., 0.0012% p. hr. The same material (pre-heated 3 hrs. at 96° C.) lost 0.113% per hr. at 130.5° C. for the first 2 hrs., and thereafter 0.0018% per hour at 106° C. for 5 hrs. The same material (pre-heated 3 hrs. at 96° C.) lost 0.021% per hr. for the first 5 hrs. at 106° C.

No. 11. The same material as No. 8 (pre-dried 3 hrs. at 96° C.) during the first 2 hrs. lost 0.454% per hr. at 130.5° C.; 3rd to 4th hr., 0.130%; 5th to 6th hr., 0.108%.

No. 12. To ascertain whether aminopiric acid—the only non-volatile product formed by hydrolysis of TNA—has any effect on the change of weight of the latter when heated, a mixture of 95% of standard TNA and 5% of aminopiric acid was heated at 130° C. The % per hour decrease was identical with that obtained with the same TNA by itself. Aminopiric acid by itself, after 4 hrs. preheating at 95° C., lost during the first 4 hrs. 0.075% per hr., and during the 5th–8th hrs. 0.048% at 130° C.

No. 13. The % per hour decrease of a mixture of 95% of TNA and 5% of aminopiric acid remained the same when the composition was finely ground to ensure intimate mixing.

#### C.—Fume (German) Test. 135° C.

No. 14. TNA was found to withstand the test (no fumes) for 60 hrs. plus.

No. 15. TNA which had been further dried on plant scale at 80° C. for 6 hrs. in a partial vacuum turned the blue litmus paper to a neutral pink (3 samples) after 34 min. without bleaching the dye. A fresh paper inserted thereafter was turned pink in 72 mins. (3 samples). The test paper did not lose its sensitiveness. The TNA gave no fumes and did not smell of acid.

TNA which had been pre-dried in the laboratory for 2 hrs. at 100° C. and then allowed to cool for 1 hr., turned the paper, at 135° C., to a neutral pink after 43 mins. (1 sample), and 63 mins. (1 sample). There were no fumes or acid smell.

<sup>11</sup> J., 1910, 132.

<sup>12</sup> Rec. Trav. Chim., 1920, 29, 163; Van Duin, Utrecht, Thesis, 1918.

<sup>13</sup> As zinc iodide test paper is more sensitive than KI paper (cf. Marshall, "Explosives," 2nd ed., p. 660) these figures are somewhat too high.

<sup>14</sup> A diagram of this test has been published in Z. ges. Schießw. Sprengst., 1919, 8, 185.

*D.—Storage test at 90° F. (32° C.)*

No. 16. Explosives for authorisation in Great Britain must withstand this test for six weeks.<sup>13</sup> TNA is on the British Home Office list of authorised explosives of January 1st, 1920, in Class 3, Division 2.

*E.—Storage test at 75° C.*

No. 17. TNA withstands this test, not only for the 48 hrs. prescribed by the International Commission for Unification of Explosives,<sup>14</sup> but for an indefinite period.

*F.—French litmus tests.*

No. 18. Moist neutral litmus paper is not reddened by TNA in 5 days at the ordinary temperature.

No. 19. Dry blue litmus paper is not reddened by TNA in 36 hrs. at 96° C.

No. 20 (test prescribed by French railways). Neutral litmus paper is not reddened by 100 c.c. of water with which 1 g. of TNA had been shaken for  $\frac{1}{2}$  hr. at 15° C.

*G.—Behaviour toward metals.*

No. 21. Experiments already recorded<sup>15</sup> have shown that TNA, whether wet or dry, does not attack steel, iron, tin, copper, brass, lead, aluminium, or zinc in 14 days at the ordinary temperature.

No. 22. TNA (moisture about 0.2%) was packed in copper caps, sealed with paraffin, and stored for 28 months at 90°–100° F. Both the TNA and the surface of the metal remained unaltered.

*H.—Behaviour towards inorganic nitrates.*

No. 23. 40 parts of TNA and 60 parts of commercial ammonium nitrate, neither of which had been specially pre-dried, were ground together and stored for seven years, under a glass jar, at ordinary temperature and in daylight, and excluding atmospheric moisture. No interaction had taken place, since the mixture was separated into its unchanged components by means of benzene or water.

No. 24. Compressed cartridges containing 50% of "yellow" TNA from an early experiment on semi-plant scale, 10% of potassium nitrate, and 40% of barium nitrate<sup>16</sup> were stored for 28 months at the ordinary temperature, with access of air moisture, and during part of this period in daylight. They remained unchanged.

*I.—Resistance to water, solubility, and hygroscopicity.*

No. 25. Wet TNA as manufactured retains about 20–25% of water on a vacuum filter, and less than 10% on a centrifugal. TNA with 20–25% of water was generally dried at a temperature rising slowly from 40° to 60° C. for about 24 hrs. at atmospheric pressure, or about 8 hrs. in a vacuum dryer. Its acidity was determined, both before and after drying, according to a method specified by two of the Allied and associated Governments, in which 10 g. of TNA is allowed to stand for  $\frac{1}{2}$  hr., with occasional shaking, in 150 c.c. of neutral water, whereafter it is filtered and twice washed with 20 c.c. of water, the united filtrates being titrated with *N*/50 sodium hydroxide, using 15 drops of a 0.5% phenolphthalein solution as indicator. The acidity obtained for standard "green" TNA generally amounted to about 0.2% calculated as  $H_2SO_4$ , and was the same before and after the drying. There is therefore no

measurable hydrolysis during drying, since this would produce aminopicric acid—a strong and non-volatile acid—which would result in a higher acidity in the dry, as compared with the wet material.

No. 26. *Exposure to own moisture on storage.* The official report states: "On a sample of TNA, Lot No. 3/16, inspected in August, 1918, for . . . I determined the acidity at that time to be 0.015%. After 9 months' dark storage of this sample, at laboratory temperature, I again determined the acidity and found it to be 0.017%. This apparent increase in acidity is within the limit of experimental error. The amount of moisture (about 0.2%) ordinarily present in TNA of regular plant manufacture does not therefore cause any decomposition of the material."

No. 27. *Acidity of TNA stored with excess of water in the absence of light.* Three tests were made as follows: 10 g. of a composite sample of ordinary green TNA was placed with 150 c.c. of neutral water in sealed rubber-stoppered bottles in a dark room and shaken occasionally and the acidity tested as described in No. 25 with the following results:—

	After	Acidity,	Increased
	1 day	..	acidity
	2 days	..	per day.
15	..	0.0193%	..
21	..	0.0231	..
15	..	0.0341	..
21	..	0.0354	..
			0.0038%
			0.0008
			0.0002

The above indicated that, in the dark, the acidity obtained with water is due to the gradual liberation of the occluded sulphuric acid. In further tests, in which TNA, as manufactured was allowed to stand with water for 6 mins., the acidity with unground TNA was found to be 0.034% and with the finely ground substance 0.063%. It is stated in the official report that these figures and the results of tests Nos. 28 and 29 show that the acidity is due to the removal of occluded sulphuric acid, since if it had been due to decomposition of the TNA the figures for "increased acidity per day" would have shown a rise or at least remained steady.

No. 28. *Prolonged exposure to excess of water in daylight.* A blend of green and yellow TNA, original acidity 0.0232%, showed the following increases of acidity: After 1st 24 hrs., 0.0209%; 24–48 h., 0.0270; 48–72, 0.0092; 72–96, 0.0256; 96–120, 0.0228; 120–168, 0.0392; 168–192, 0.0110; 192–216, 0.0147; 216–264, 0.0287; 264–312, 0.0140; 312–336 h., 0.0094%. Average increase of acidity per 24 hrs., 0.0159%. Two other samples showed an average increase of acidity per 24 hrs. of 0.0163% and 0.0139%. The daily variation in the increase of acidity appeared to depend somewhat on the changes of temperature. That the TNA, through the action of water at ordinary temperature, does not undergo any change that would affect its quality is demonstrated, the official report adds, by the fact that the three samples after the above tests had m.p. 212.5°, 213.0°, and 213.0° C. respectively.

No. 29. The sample of TNA which had been stored with excess of water for 13 days without complete exclusion of light was dried at 60° C. and then heated for one hour at 100° C.

(a) Abel test at 65.5° C.; 3 specimens. The test paper was not coloured in 1 hr. 50 min., whereupon the test was discontinued.

(b) Heat test at 136°–140° C.; 4 specimens. The first indication on the litmus paper of a change from blue to neutral pink appeared after 17 minutes. After an additional 58 mins. (viz., a total heating of 1 hr. 15 min.) the papers had entirely changed to a neutral pink, with the exception of a strip on the margin of each, which had originally been more intensely blue than the rest of the paper, and which

<sup>13</sup> Dupré, see "Arms and Explosives," 1915, p. 129.

<sup>14</sup> Orig. Comm., Int. Cong. Appl. Chem., 1912, 28, 311.

<sup>15</sup> Z. Ges. Schless. u. Spreng., 1913, 8, 185.

<sup>16</sup> These cartridges were exhibited by W. Macnab in his lecture on Explosives (loc. cit.).

remained blue. The TNA gave no fumes and did not smell of acid.

These tests are, states the report, in the case of the Abel test as good as, and in the case of the 136°–140° C. test better than the results obtained with the same sample before storage with water.

No. 30. Prolonged exposure to a smaller amount of water in daylight, at ordinary temperature. 10 g. of TNA with 4 g. of water were left for 33 days, then washed and filtered. The filtrate showed 0.054% of acid. 10 g. of TNA which had been shaken with water for a few minutes, then washed, gave 0.03% of acid (*cf.* No. 27).

No. 31. Prolonged exposure to excess of moisture in partial daylight. TNA as used in test No. 8, but with 10% of water and an unusually large amount

Ten weeks' ordinary moist storage (80% saturation):—

(Gain in weight calculated on original weight.)

Week.	Gain in wt. (g.).	Lead block, c.c.	Week.	Gain in wt. (g.).	Lead block, c.c.
1st ..	0.0015 ..	13.0	6th ..	0.0030 ..	12.8
2nd ..	0.0019 ..	12.9	7th ..	0.0030 ..	12.6
3rd ..	0.0021 ..	12.85	8th ..	0.0030 ..	12.6
4th ..	0.0035 ..	13.0	9th ..	0.0030 ..	12.6
5th ..	0.0025 ..	12.9	10th ..	0.0030 ..	12.6

The keeping qualities of TNA detonators when exposed to moisture at ordinary temperature are thus quite satisfactory. This was confirmed by lead-plate tests carried out on No. 6 detonators, after a year's ordinary and a year's hot-moist storage at 40° C., as shown by the accompanying illustrations.



of sulphuric acid remaining after perfunctory washing in the plant, was left in a stoppered amber bottle, on an open shelf, for 35 months. When dried thereafter—without any previous washing—its melting point, when compared, by simultaneous heating, with that of a sample of the same material which had been carefully washed and dried a few days after manufacture, as well as its appearance (colour etc.) were found to be unchanged.

No. 32. *Solubility of TNA in water.* One g. of finely divided TNA was shaken for ½ hr. with 100 c.c. of water at 13° C. in order to remove traces of mineral acid and soluble impurities introduced by the factory water-wash. The material was filtered, washed, and then left for two hours in one litre of water, with frequent shaking. After filtering the water was evaporated at 100° C., a little ammonia having been added to retain all transformation products. The solubility was thus found to be approximately 1:50,000 at 13° C.

Even in solution TNA does not react with water as readily as appears to be commonly supposed<sup>11</sup>:

No. 33. 20 g. of TNA was dissolved in 225 c.c. of acetone at about 30° C., filtered, and stirred into 400 c.c. of water at ordinary temperature. About 90% of the TNA was thus recovered; the rest remained in the mixed solvent, also unchanged, since, even after an hour, the solution gave no nitrous acid reaction with potassium iodide test paper.

No. 34. *Hygroscopicity.* This has been given<sup>12</sup> as 0.12% for a material made on a small scale. This figure approximately holds good for the standard ("green") commercial material. A sample which had been plant-dried to 0.49% of moisture was completely laboratory-dried at 95° C., and then exposed to moist air until its weight became constant, when it was found to contain 0.13% of moisture. A more finely divided ("yellow") TNA became constant after 32 days, with 0.19% of moisture.

No. 35. *Storage of TNA in detonators.* The detonators were manufactured with 0.3 g. of 80% fulminate and 0.4 g. of TNA per detonator, at a pressure of 176 lb.

J.—*Consideration of tests Nos. 1 to 35 in relation to each other and to the behaviour of other nitro-explosives with a mobile nitro-group [tetryl, nitroglycerin (nitroglycol), nitrocellulose (nitrostarch)].*

According to the bulk of expert opinion (*cf.* Guttman,<sup>13</sup> Nathan,<sup>14</sup> Thomson,<sup>15</sup> Reid,<sup>16</sup> Cullen,<sup>17</sup> Aspinwall,<sup>18</sup> Spica,<sup>19</sup> Barthelmy,<sup>20</sup> Sy,<sup>21</sup> Lenze,<sup>22</sup> Escalas,<sup>23</sup> Wilf<sup>24</sup>), the Abel heat test records, in the first place, not the degree of stability, but the degree of purity of an explosive. When a given impurity is known to lower the heat test of a given explosive and at the same time to cause its progressive decomposition, then the Abel test becomes indirectly a measure of the stability of a particular explosive as affected by a particular impurity. It therefore does not necessarily indicate the relative stability of samples of the same explosive contaminated by different impurities; still less does the Abel test claim, to quote Cullen,<sup>17</sup> to measure the relative stability of different explosives.

Nevertheless, if it is often impossible to draw definite conclusions from a bad Abel test, a good test, provided that no foreign substance which would mask oxides of nitrogen is present, necessarily indicates a corresponding degree of stability; moreover, the Abel tests usually obtainable from good commercial grades of various explosives enable certain conclusions to be drawn as to their relative stability if the effect of impurities normally present on both the test and the stability is taken into consideration.

According to test No. 1, Van Duin and Van Lenoep obtained a TNA which would evolve rather less than 0.01% of NO<sub>2</sub> in 115 years at 32° C. (89° F.) and in 37 years at 50° C. (122° F.).<sup>25</sup> In the preparation of this sample potassium nitrate and

<sup>11</sup> J., 1901, 9-12.

<sup>12</sup> J., 1902, 688.

<sup>13</sup> Rep. of Int. Comm. on "Unification of stability tests of explosives," 7th Int. Cong. Appl. Chem., 1900; Sec. II, 179.

<sup>14</sup> *Ibid.*, p. 174.

<sup>15</sup> U.S. Arch. J., 1903, 20, 138.

<sup>16</sup> Z. ges. Schless.-u. Spreng., 1909, 4, 303.

<sup>17</sup> "Explosivstoffe," Vol. 2, 179; "Nitroglycerin und Dynamit," 151, 1908.

<sup>18</sup> Z. angew. Chem., 1901, 14, 775.

<sup>19</sup> *Loc. cit.*

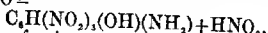
<sup>20</sup> These authors (see also Van Duin and Van Lenoep, *Rec. Trav. Chim.*, 1917, 37, 114; Van Duin and Brackmann, *Chem. Weekbl.*, 1919, 19, 501) and also Stettbacher, base on these results, and on these alone, their allegation that TNA shows "rapid decomposition." Obviously they failed to realise the quantitative meaning of their tests.

<sup>21</sup> Test No. 33 proves that Van Duin (*Rec. Trav. Chim.*, 1917, 37, 117; 1920, 39, 150) is mistaken when he makes water responsible for the instantaneous reaction of TNA with sodium acetate in aqueous acetone at ordinary temperature which had been previously described by the present author. Besides, numerous other basic reagents react similarly in the total absence of water.

<sup>22</sup> Z. ges. Schless.-u. Spreng., 1913, 9, 185.

acetic acid had been used, both of which lower the heat test of TNA and are never employed in its manufacture. It is therefore not surprising that, on a commercial scale and without any purification other than water-washing, a TNA is obtained which, when properly dried on a plant scale, gives still better results, such as the official tests No. 2.

Nos. 2 and 3 show the effect of moisture on the Abel test. Attention had already been called<sup>11</sup> to the fact that TNA—which, as evidenced by Nos. 21, 25, 30, and 31 and the official tests No. 28 to 29 and 22 is not affected by moisture under any conditions arising during its manufacture, storage, and use—at more elevated temperatures slowly reacts with water<sup>12</sup> according to the equation:



producing the trace of nitrous acid required to colour the test paper. If at the ordinary temperature the action of even large amounts of water becomes immeasurably small, this must be ascribed not alone to the temperature coefficient of this reaction itself, but to its product with the temperature coefficient of the solubility of TNA in water, which solubility, at the ordinary temperature, is practically nil (see No. 32).

That the quality of TNA does not suffer by the action of moisture during the heat test (No. 2) is due to the fact that the permanent product of the reaction, aminopieric acid, does not affect the stability of TNA (Nos. 12 and 13).

The gas evolution tests at 120° C. are in good agreement with the loss of weight at 130° C., for a

perature coefficient<sup>13</sup> of about 2 for 5° C. Tests No. 3 prove that the gas contains only traces of nitrogen oxides (initially about 1%). These include nitrous and nitric acids occluded in the crystals and NO<sub>2</sub> produced by the action of water vapour derived from the decomposition of oxalic acid, and their gradual elimination causes the rate of evolution of NO<sub>2</sub> to fall off (cf. No. 3, f and g, and, for elimination of occluded NO<sub>2</sub> by storage, No. 2, c and d), until it finally reaches a figure—exceedingly small even at 130° C., and too small for observation at 65.5° C.—representing the actual decomposition of TNA.<sup>14</sup>

Where even traces of occluded impurities, such as certain acids, lower the stability, as with tetryl,<sup>15</sup> the gas evolution increases with the size of the crystals<sup>16</sup>; conversely, actual decomposition of a pure material appears to decrease with increasing size of crystals.<sup>17</sup> Tests Nos. 5 and 6, and 12 and 13, prove that the initial gas evolution of commercial TNA remains unaffected by the size of the crystals. On the other hand, the evolution of NO<sub>2</sub> is higher for ground than for ordinary TNA, even after the effect of steam from oxalic acid decomposition has been practically eliminated by 22 hours' pre-heating at 130° C. Both these facts, again, are explained when the bulk of the initial evolution of total gas is ascribed to removal of an impurity—oxalic acid—which, like aminopieric (cf. Nos. 12 and 13) or sulphuric acid, does not affect the stability of TNA. In the following table, the gas evolution from TNA and other explosives containing a mobile nitro or nitrate group is compared:—

	Total gas at 120° C. (c.c. per g. per hr.)			NO <sub>2</sub> at 120° C. (mg. per g. per hr.) 1st 8 hrs.	Effect of product of hydrolysis on gas evolution.
	First 2 hrs.	First 40 hrs.	100th to 200th hour.		
TNA (unrecryst., commercial) .. .. .	0.425	0.089	0.005 <sup>18</sup>	about 0.0007	5% aminopieric acid no effect.
Tetryl (unrecryst., comm.) .. .. .	0.07	>0.135	—	—	—
.. (recryst., British Service) .. .. .	0.020 <sup>19</sup>	0.022 <sup>20</sup>	0.75 <sup>21</sup>	about 0.01 <sup>22</sup>	10% pieric acid causes 14% initial increase. <sup>23</sup>
Guncotton (good quality, Brit. Service) .. .. .	—	0.366 <sup>24</sup>	—	about 0.01 aver. <sup>25</sup>	—
Nitroglycerin <sup>26</sup> .. .. .	—	3.65 <sup>27</sup>	—	5 <sup>28</sup>	—
Amatol (50% crude TNT, 50% pure ammonium nitrate) .. .. .	—	0.015 <sup>29</sup>	—	—	—

gas composition of equal volumes of water vapour, carbon monoxide and dioxide (see below), weighing about 1½ mg. per c.c. at 0° C. and 760 mm., a temperature coefficient of approximately 2 per 5° C., and corresponding stages of the evolution. The same temperature coefficient is obtained when gas evolution tests Nos. 4 and 7 are compared with each other (i.e., practically the same evolution at 100° C. in first 32 hours as at 120° C. in first 2 hours). There is always a relatively substantial initial evolution or loss of weight, indicating the removal of traces of an impurity, followed by a smaller evolution at a decreasing rate. The impurity is very probably oxalic acid, for the following reasons: The quantitative course of TNA nitrations indicates the formation of substantial amounts of oxalic acid, which is only sparingly soluble in strong sulphuric acid. When nitration is carried out increasingly under conditions (which are well known) causing decomposition of oxalic acid, catalysed by sulphuric acid, the apparent colour of TNA (before drying) changes from yellow to greenish, the occlusion (see under II.) in the crystals decreases, and their gas evolution and loss of weight decrease. Oxalic acid, catalysed by traces of sulphuric acid, is known to decompose into carbon monoxide and dioxide and water at the temperatures of the tests, and with the above tem-

The NO<sub>2</sub> figures explain the difficulty in obtaining a commercial tetryl which will give as good an Abel test as TNA, even after recrystallisation; also the fact that TNA does not evolve brown fumes at 135° C. in 60 hours, whereas good American tetryl was found to do so in 40–60 minutes—thus indicating inferior stability even after allowing for the fifty-fold increase caused by the liquid state<sup>30</sup>

<sup>11</sup> Cf. Bredig, Z. Elektrochem., 1906, 12, 159.

<sup>12</sup> The approximate accuracy of calculations, by Robertson and Smart's formula, of very low NO<sub>2</sub> concentrations from Abel tests is evidenced, for instance, by the good agreement of figures thus calculated for guncotton at 71° C. with Robertson and Napper's actual determinations of NO<sub>2</sub> at 135° C. (J. Chem. Soc., 1907, 91, 769), taking Robertson's temperature coefficient of 1.9 for 5°.

<sup>13</sup> Farmer, J. Chem. Soc., 1920, 117, 1603.

<sup>14</sup> Hinshelwood and Bowen, Phil. Mag., 1920, 40, 569.

<sup>15</sup> Calculated from gas evolution at 140° C., as loss of weight tests at 130° C. include traces of volatile solid matter which count where decrease is small.

<sup>16</sup> Figures obtained by different observers with different regular Service samples.

<sup>17</sup> Calculated from Farmer's test at 125° C. (loc. cit.).

<sup>18</sup> From figures by Robertson (loc. cit.); Robertson and Napper, J. Chem. Soc., 1907, 91, 783.

<sup>19</sup> The NO<sub>2</sub> evolution of NG can be calculated for ordinary temperature by Robertson's temperature coefficient (2.2 for 5° C.). In view of this and the comparatively enormous decomposition of NG at 120° C., Stettbacher's remark (Schweiz. Chem.-Zeit., 1919, 1, 95) that NG, while less stable than TNA at 70° C., was more stable at ordinary temperature, is absurd. Van Damm's (Rec., 1919, 39, 103) corresponding reference to the absence of acidity in Sobrero's preserved sample of NG overlooks the precautions taken to remove acidity.

<sup>20</sup> Robertson (J. Chem. Soc., 1921, 119, 1; 1909, 95, 1241).

<sup>21</sup> Robertson, loc. cit.

<sup>22</sup> Farmer, J. Chem. Soc., 1920, 117, 1603.

<sup>23</sup> Flürschheim, Z. ges. Schless.-u. Spreng., 1913, 8, 185.

<sup>24</sup> Whether the same is mixed with the TNA, or derived from the wet test paper.

of tetryl at 135° C. The above tests, moreover, afford support for the proposals of the International Commission on Unification of Explosives, Barthelmy, Lenze, and others, to use a storage-fume test at more or less elevated temperatures as the standard stability test, since loss of weight, gas evolution, and trace tests alone are seen to be misleading unless the nature of the change is taken into consideration. Taken in conjunction with a fume test, however, the other stability tests bring out the following crucial fact concerning the relative stability of TNA, with its mobile nitro-group attached to an aromatic ring carbon atom, compared with explosives containing a mobile nitro-group attached to oxygen—such as nitrocellulose, nitroglycerin, or nitrostarch—or to nitrogen, *e.g.*, tetryl:—*Whereas with TNA traces of certain impurities, which are either normally present or else produced in some conditions by the mobile nitro-group, may affect the Abel test, but do not affect the stability, with nitrocellulose, nitroglycerin, tetryl, etc., they generally affect both the test and the stability.* TNA is accordingly intrinsically more stable than any of these materials, and this becomes particularly obvious when old industrial products, and not specially purified samples, are compared.

"Instability" (*i.e.*, tendency to intramolecular, spontaneous decomposition) must not be confounded with "reactivity" (*i.e.*, tendency to inter-molecular reaction with other substances). Thus the reactivity of a nitro-group contiguous to two other nitro-groups—which obtains in TNA—is also to be found in 2,3,4-trinitrotoluene, but less in 3,4,6-, and still less in 2,4,6-trinitrotoluene. Notwithstanding this, Farmer<sup>40</sup> found the gas evolution to be no greater for the 2,3,4- than for the 2,4,6-, and less than for the 3,4,6-compound.

Even reactivity depends essentially on solubility, and the practical insolubility of TNA in water at usual temperatures, together with the lack of autocatalysis by impurities, no doubt largely accounts for the fact that, during periods of storage of TNA, with its own moisture,<sup>41</sup> ranging from 7 to 17 years, no change either in appearance, composition, or melting point has been observed, even with the first samples—some of which were acid or otherwise impure.

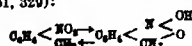
#### K.—Resistance to light.

No. 36. Samples of TNA of varying preparation and purity have been exposed to daylight in ordinary glass bottles on open shelves for periods ranging from 7 to 17 years. In no case has there been any change in appearance or otherwise. Certain other explosives, *e.g.*, nitroglycerin,<sup>42</sup> hexanitrodiphenylamine,<sup>43</sup> and TNT,<sup>44</sup> are, on the other hand, affected by daylight.

<sup>40</sup> Farmer, *loc. cit.*  
<sup>41</sup> Farmer (*loc. cit.*) states: "On account of the instability of the nitro-amino group, this compound (tetryl) . . . may undergo deterioration on storage if not completely purified."  
<sup>42</sup> J. Chem. Soc., 1920, 117, 1442.

<sup>43</sup> In actual practice, whether used in blasting cartridges, fuses, shells, bombs, torpedo heads, or elsewhere, the material will necessarily be stored in the dark and in a compressed or cast form. As shown by the tests on exposure to water, moist behaviour to metals, etc., there is in such conditions no change, even with loose material offering a large surface of reaction, and with moisture in excess of the 0.12-0.2% normally present.

<sup>44</sup> Compare Gody, "Matières Explosives" (1907).  
<sup>45</sup> Carter, Z. ges. Schless.-n. Spreng., 1913, 3, 251.  
<sup>46</sup> Cf., *e.g.*, Molinari and Quartieri, Z. ges. Schless.-n. Spreng., 1913, 3, 405, 432. Even the purest TNT is turned green by daylight, a change which is, no doubt, analogous to the photochemical transformation of *o*-nitrotoluenes to *o*-nitrosobenzic acid. This reaction as well as the condensations of *o*- and *p*-nitrotoluenes to stilbenes, etc., and some new observations to be published later, may be attributed to tautomerism (in analogy with the author's bridge formula for primary and secondary nitro-groups; J. prakt. Chem., 1902, 88, 161, 329):



<sup>47</sup> Z. ges. Schless.-n. Spreng., 1913, 3, 185.

#### L.—Resistance to fire.

The flash point of TNA had been found<sup>45</sup> by a British firm of explosives manufacturers to be 220° C., compared with 186° C. for tetryl. The temperatures of explosion of 247°—237° C. (*corr.*) for TNA and 196°—187° C. (*corr.*) for tetryl have since been given by Van Duin and Van Lennep.<sup>46</sup>

No. 37. Some experience is now available on the effect of fire on large quantities of TNA. Once a fire, caused by a diphenylamine autoclave, extended to large vats in which about 16,000 lb. (dry weight) of TNA containing 20 to 25% of moisture was stored. Though the terrific heat of the burning TNA twisted the steel girders of the building into almost unrecognisable shapes, the TNA merely flashed, but did not explode.

No. 38. Later, a TNA packing room was, in the absence of the workers, set on fire by a time fuse; several thousand pounds of dry TNA, nailed down in cases ready for shipment, again flashed without explosion.

#### M.—Resistance to shock.

Comparative drop-weight tests are not always reliable,<sup>47</sup> and the results previously given<sup>48</sup> have not been confirmed. Later experience proves that TNA is slightly more sensitive than picric acid, and slightly less sensitive than tetryl. Thus, under strictly comparable conditions, the relative insensitiveness to mechanical shock is expressed by the figures:—

	TNT.	Picric acid.	TNA.	Tetryl.
No. 39 (Van Duin and Van Lennep) <sup>49</sup> :				
2 kg., maximum height in cm. for no detonation out of 6	..	< 60	< 60	< 60
Minimum height in cm. for 6 detonations out of 6	..	< 60	54.5	52
No. 40 (R. Robertson) <sup>50</sup> : Relative figures ..	115	100	68	70

In view of these results, which agree with general experience, the impression conveyed by J. Marshall<sup>51</sup> (of the Du Pont Powder Company) that TNA was slightly more sensitive to shock than tetryl is obviously incorrect.

No. 41. A. P. Howell's<sup>52</sup> pendulum friction tests afford a comparison with some other explosives:—

Steel shoe (with 44 lb. added.) 1.5 m. swing.		Steel shoe (with 44 lb. added.)	TNA.		NG-dynamite 40%	
			Tested.	Passed.	Tested.	Passed.
Passed TNT. etc.	Failed Tetryl. TNA. 50/50 Amato.	1.0 m. Swing.	1	0	1	0
		0.5 m. Swing	1	1	1	0
		2 m. Swing (with aloxite cloth)	1	1		

No. 42. Admixture of very little paraffin reduces the sensitiveness of TNA to mechanical shock below that of TNT; *e.g.*, with 5% of paraffin uniformly distributed throughout the material, with 2 kg.,<sup>53</sup> the maximum height for no detonation in 5 out of 5 for TNA was 7 in., TNT 11 in., and TNA 95%, paraffin 5%, 28 in.

<sup>48</sup> Rec. Trav. Chim., 1920, 39, 170.

<sup>49</sup> Compare R. Robertson, J. Chem. Soc., 1921, 119, 16.

<sup>50</sup> Z. ges. Schless.-n. Spreng., 1913, 3, 165.

<sup>51</sup> Rec. Trav. Chim., 1920, 39, 174. Van Duin's assertion (with K. Brackmann, Chem. Weekblad, 1919, 16, 601) that tetranitro compounds, while stronger, are more sensitive to shock than tetryl is thus, as regards TNA, disproved by his own results.

<sup>52</sup> J. Chem. Soc., 1921, 119, 18.

<sup>53</sup> J. Ind. Eng. Chem., 1920, 12, 336.

<sup>54</sup> U.S. Bureau of Mines, Tech. Paper 234, 1919.



No. 43. The Trauzl block "strength" of TNA is not much reduced by such mixtures with 5% or less of paraffin, but is materially affected by 10% of paraffin; *e.g.*, the Trauzl block net expansion (c.c. per 10 g.) of pure TNT was 216, TNA 315, TNA with 5% paraffin 235 and 295, and with 10% paraffin 205 and 225.

No. 44. Six-pdr. projectiles loaded with a mixture of 95% TNA, 5% paraffin (Mixture "B"), were fired through a 24 in. steel plate: the respective weights were 6.05 and 5.94 lb., and the striking velocities 2381 and 2382 f.s. In each case the impact was normal and the projectile penetrated the plate. In the first case the projectile was recovered whole and undeformed, the burster charge remaining entirely inert. In the second case the projectile was recovered broken transversely, in two pieces just forward of the band score. Here again the bursting charge remained entirely inert.

health of operators or users. It is well known that other nitro-explosives<sup>41</sup> either affect the internal organs, notably TNT<sup>42</sup> before it is completely purified, NG<sup>43</sup>, picric acid,<sup>44</sup> tetryl<sup>45</sup>; or else the skin, *e.g.*, hexanitrodiphenylamine,<sup>46</sup> trinitroanisole.<sup>47</sup>

#### V.—POWER AND SENSITISING PROPERTIES.

The outstanding feature of TNA is its enormous shattering power or brisance (B), which results from its combining a high velocity of detonation (*v*) with great density (*d*) and great energy (E), as expressed by Herlin's<sup>48</sup> formula:  $B = E \cdot d^{1/3} \cdot v^2$ , wherein K is a constant. The energy E or "strength," measured by the pressure of the explosion gases, is proportional<sup>49</sup> to the volume of the explosion gases, per gram of explosive, at the temperature of explosion and at atmospheric pressure.

No. 47. In the following table, column V gives the relative Trauzl block expansions of a number

Solid high explosives (other than mixtures).	I. Temperature of explosion, °C.	II. Gas vol. at 1° in litres per gram.	III. Relative "strength" calculated.	IV. Relative "strength," reduced Trauzl block enlargement per 10 g.	V. Relative "strength," actual enlargement of Trauzl block per 10 g.	VI. Maximum density.	VII. Relative "strength," reduced Trauzl block enlargement per 6 c.c. of explosive at maximum density.
TNA .. .. .	3238 <sup>51</sup>	10.55 <sup>51</sup>	395 <sup>51</sup>	370	415 <sup>51</sup>	1.9	421
Tetryl .. .. .	3590 <sup>52</sup>	11.4 <sup>52</sup>	427 <sup>52</sup>	342	375 <sup>52</sup>	1.73 <sup>52</sup>	355
Trinitrobenzene <sup>53</sup> .. .. .	—	—	—	331	264	1.67 <sup>53</sup>	335
Hexanitrodiphenyl sulphide <sup>54</sup> .. .. .	—	—	—	228	355	1.70 <sup>54</sup>	335
Hexanitrodiphenylamine <sup>55</sup> .. .. .	—	—	—	326	352	1.71 <sup>55</sup>	334
Picric acid <sup>56</sup> .. .. .	2569	8.727	328	310	330	1.75 <sup>56</sup>	320
Guncotton (dry) <sup>57</sup> .. .. .	—	—	—	307	325	1.634 <sup>57</sup>	300
Trinitrochlorobenzene <sup>58</sup> .. .. .	—	—	—	304	322	1.77 <sup>58</sup>	323
Trinitroanisole <sup>59</sup> .. .. .	—	—	—	300	322	1.65 <sup>59</sup>	297
Nitrostarch <sup>60</sup> .. .. .	2205.7	8.163	306	290	305	—	—
Trinitrocresol <sup>61</sup> .. .. .	—	—	—	287	301	1.60 <sup>61</sup>	291
TNT <sup>62</sup> .. .. .	2217	6.764 <sup>62</sup>	253 <sup>62</sup>	265	274 <sup>62</sup>	1.65 <sup>62</sup>	262

No. 45. Shell fragmentation obtained with above mixture. The report says:—"4-in. Common projectile 1. Weight empty, with base and fuze 28.82 lb. Bursting charge, 2.03 lb. Mixture "B" (tamped) plus 63 g. loose TNA as a booster. Standard 8 dynamite cap and Bickford fuze fitted through fuze plug. Recovered 276 fragments weighing 11.66 lb. Weight of largest fragment 29 lb. (base plug); smallest fragment less than 2 grains. Average weight of fragment 0.042 lb. Calculated number of fragments 663." In these tests the material was only hand-tamped, otherwise a substantially higher loading density would be obtained.

No. 46. Behaviour of TNA towards concussion by an explosive wave. 5000 lb. of TNA, packed in open cases, was stored in a magazine of galvanised iron, protected by an earth barricade. A packing room a few yards beyond the barricade was blown up, leading to an explosion of about 800 lb. of dry TNA, which precipitated a rock on a railroad cutting about five miles away, partially demolished the barricade, warped the magazine, and threw about the TNA cases, but the 5000 lb. of TNA remained intact. It may be added that, as far as the author is aware, this is the only explosion of TNA which has so far occurred in a TNA plant. It was attributed by the authorities to sabotage.

#### IV.—PHYSIOLOGICAL PROPERTIES.

TNA shares with nitrocellulose (nitrostarch etc.) the distinction of having no injurious effect on the

of explosives. They have been referred to tetryl= 375 c.c. by means of Kast's<sup>63</sup> graphical reducing method for elimination of the excess of expansion due to progressive weakening of the block, the reduced figures being given in col. IV; col. VII contains the corresponding reduced Trauzl block figures calculated for equal volumes (6 c.c.) at maximum densities.

- <sup>41</sup> Compare Van Duin, *Chem. Weekblad*, 1919, 16, 202.  
<sup>42</sup> Comp. Hamilton, *J. Ind. Eng. Chem.*, 1916, 8, 235; Casselmann, *Z. ges. Schiess- u. Spreng.*, 1918, 13, 245; Koelsch, *ibid.*, 1917, 12, 133; Berndtgen, *J. Chem. Industrie*, *ibid.*, 1917, 12, 62; Colver, "High Explosives," 206 (1918).  
<sup>43</sup> Cf. Swin (Du Pont Powder Co.), U.S.P. 1,309,553.  
<sup>44</sup> Cf. Badeloff, *Z. ges. Schiess- u. Spreng.*, 1907, 2, 4; Koelsch, *loc. cit.*; Colver, *loc. cit.*, p. 243; Hamilton, *loc. cit.*  
<sup>45</sup> Cf. Colver, *loc. cit.*, p. 372; Escalas, "Nitro-Explosives," p. 194 (1915).  
<sup>46</sup> Cf. Marshall, "Explosives," II. Ed., 273; Carter, *Z. ges. Schiess- u. Spreng.*, 1913, 8, 251.  
<sup>47</sup> Cf. Hamilton, *loc. cit.*; Casselmann, *loc. cit.*; Koelsch, *loc. cit.*  
<sup>48</sup> *Z. ges. Schiess- u. Spreng.*, 1914, 9, 236.  
<sup>49</sup> E. Waller, *J. Ind. Eng. Chem.*, 1918, 10, 144.  
<sup>50</sup> *Z. ges. Schiess- u. Spreng.*, 1920, 15, 173, 192.  
<sup>51</sup> E. Waller (*J. Ind. Eng. Chem.*, 1918, 10, 144) who has calculated temperatures of explosion by Mollard and Le Chatelier's formula (*Z. ges. Schiess- u. Spreng.*, 1920, 15, 173, 192).  
<sup>52</sup> Temperature for tetryl calculated by Kast should lead to the higher of the two temperatures given for TNA.  
<sup>53</sup> Kast, *loc. cit.*  
<sup>54</sup> G.P. 260,571.  
<sup>55</sup> This relative figure for TNT is, according to the author's experience, generally obtained with a No. 8 detonator, and about 0.255 with a No. 6. TNA and tetryl are little affected by the size of the primer.  
<sup>56</sup> According to pressure gauge results obtained by Cruikshank (*J. Franklin Inst.*, 1919, 189, 607) the "strength" of hexanitro- diphenylamine would exceed that of tetryl by 45%. This is not only contrary to general experience, but theoretically impossible.  
<sup>57</sup> Bichel, "Testing Explosives," Table III., value referred to TNT.  
<sup>58</sup> Escalas, "Schirbaumwolle."

<sup>44</sup> The figures of Nos. 39 and 42 again show that only drop-weight tests carried out under identical conditions are comparable.

As to the relative "strength" of nitroglycerin as such, the available data are contradictory. According to the calculations of Waller,<sup>78</sup> by Mallard and Le Chatelier's formula, the "strength" of NG, with an explosion temperature of 3158° C., would be only 332, compared with 395 for TNA; the same proportion would follow from Hofwimmer and Haackel's<sup>79</sup> calculations for blasting gelatin. The specific heat of gases above 2000° C. is, however, uncertain; accordingly, Kast<sup>80</sup> calculates an explosion temperature of 4250° C. for NG, and 3370° C. for tetryl, which would give relative "strengths" of NG and TNA of about 447 and 427. Comey and Holmes<sup>81</sup> found the ballistic pendulum recoil for 6.15 g. of blasting gelatin and 10 g. of TNT to be identical ( $\approx 1:62:1$ ), corresponding to a ratio of "strength" for NG and TNT of about 1:53:1; Kast's Trauzl block figures, reduced by his graphical method,<sup>82</sup> give the—incorrect (see note <sup>78</sup>)—ratio 1:58:1 for NG:TNT, and Bichel's<sup>83</sup> "Druckmesser" pressures for blasting gelatin and TNT (1:46:1) about 1:4:1 for NG:TNT. On the other hand, the ratio of TNA:TNT, according to Waller's theoretical figures, would be 1:55:1; according to average Trauzl block effects, reduced by Kast's method,<sup>84</sup> 1:4:1; and by comparative pendulum swings<sup>85</sup> 1:46:1. The ratio of the "strength" of NG to that of TNA therefore lies between the limits 0.95:1 and 1:1:1.

Concerning the relative "strength" of the amatols, Crawshaw<sup>86</sup> gave the maximum theoretical pressures, when detonated in their own volume, at 1.6 density, as 10,978 kg. per sq. cm. for an 85/15 amatol, 9851 for TNA, and 9486 for tetryl. Quite apart from the general inaccuracy of "Druckmesser" determinations,<sup>87</sup> Crawshaw had to use a "booster" of 24 g. of tetryl to detonate the amatol, deducting from the result the pressure given by the "booster" alone. He overlooked the fact that the mixture of an amatol with excess of oxygen, and tetryl with a deficit, must give a pressure substantially in excess of the added pressures of the ingredients. Crawshaw's figures are, moreover, theoretically impossible. Not even the gas volume of an 80/20 amatol,<sup>88</sup> with its almost ideal oxygen balance, could produce Crawshaw's relative pressures, even if its temperature of explosion were not very much lower than that of TNA due to its somewhat lower heat of explosion and the composition of its gases (about 60% of which, as compared with about 20% for TNA, are water vapour and carbon dioxide with a high average specific heat). Thus Waller<sup>89</sup> gives for an 78.73/21.27 amatol the temperature of explosion as 2310° C. (TNA=3238°), and its pressure ("strength") as 8.2622 (TNA=10.55).<sup>90</sup>

<sup>78</sup> *loc. cit.*

<sup>79</sup> Z. ges. Schless.-u. Spreng., 1918, 13, 175.

<sup>80</sup> *loc. cit.*

<sup>81</sup> Comm. 8th Int. Cong. Appl. Chem., 1912, 25, 217.

<sup>82</sup> *loc. cit.* His reduced figures for NG and TNT (485 and 280) are erroneous and should, by his own diagram, read "435" and "275."

<sup>83</sup> "The ratio of the 'strength' of NG, blasting gelatin, etc., to that of solid explosives, as found in the Trauzl block, is invariably too high. For solid explosives are tested as loose powders, with a low rate of detonation (about 5000 m. per sec. for aromatic high explosives at a density of 1.0) corresponding to the low density of loading; the results are comparable with each other, but not with nitroglycerin, which is tested at a density of 1.6, with a rate of detonation of above 7000 m. per sec.; such a large difference in the rate of detonation results in an additional enlargement of the cavity."

<sup>84</sup> "Testing Explosives" (1905) tables.

<sup>85</sup> See above.

<sup>86</sup> Z. ges. Schless.-u. Spreng., 1913, 8, 185.

<sup>87</sup> J. Franklin Inst., 1919, 189, 607.

<sup>88</sup> Comey and Holmes, *loc. cit.*; this is further confirmed by Crawshaw's exceedingly inaccurate value for hexanitrodiphenylamine (see above).

<sup>89</sup> Cf. Robertson, J. Chem. Soc., 1921, 119, 25.

<sup>90</sup> *loc. cit.*

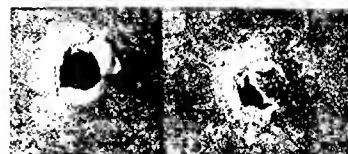
<sup>91</sup> An American 80% ammonium nitrate explosive, tested in the ballistic mortar by Comey and Holmes (*loc. cit.*), was found to possess 73% of the strength of blasting gelatin; it is therefore about equal to 75% guhr dynamite.

Not only is an 85/15 amatol substantially weaker than TNA at equal pressures, but there is also little to be gained by comparing relative theoretical pressures, up to 1.6 density, of an amatol which Crawshaw could only detonate with a booster, even at a density of 1.02 (with a theoretical pressure, even based on his figures, of only 7000 kg. per sq. cm.), with an explosive like TNA which can be detonated by an ordinary cap up to its maximum density (with a theoretical pressure, again according to Crawshaw, of about 11,500 kg. per sq. cm.).

As to the relative "strength" of tetryl, its inferiority to TNA by about 10%, as measured in the ballistic pendulum,<sup>91</sup> agrees with Waller's theoretical and experimental figures and with innumerable—partly official—Trauzl block tests. It is therefore obvious that the pendulum results of Cope,<sup>92</sup> recording for NG:TNA:tetryl:TNT the pressure ratios 14:12:1:12:1:10, cannot be correct.

Thus taking equal weights, the energy (E) of TNA approaches that of NG, and exceeds the energy of other explosives. For equal volumes<sup>93</sup> at maximum densities it exceeds all explosives. For its maximum density (d) (see under II) surpasses that of NG (1.6) by about 18%.

For its velocity of detonation (v in the brisance formula) no accurate comparative figure at equal densities are at the moment available, but it has been definitely established that it would exceed those of tetryl or nitroglycerin<sup>94</sup> by several hundred metres per second at equal densities, and exceeds them still more at maximum densities. In consequence the "brisance" of TNA is not reached by an equal weight, and still less by an equal volume at high density, of any other explosive.



TNA.

Tetryl.

No. 48. The following photographs, obtained by Stettbacher,<sup>95</sup> illustrate the superiority as regards brisance of TNA over tetryl when 3 g. of each were detonated on iron plates 3 mm. thick. Elsewhere<sup>96</sup> Stettbacher states that TNA in respect of its velocity of detonation far surpasses all other nitro-compounds, that the difference between its brisance and that of TNT is perhaps as great as between TNT and dinitronaphthalene; the difference between its brisance and that of tetryl at least as great as between tetryl and TNT; and that its explosive properties undoubtedly surpass those of all other explosives.

No. 49. An idea of the comparative brisance of TNA and TNT is conveyed by the following test:—A bar of wrought iron 5 in. wide, 4 in. thick, was laid on steel supports 154 in. apart. A paper cartridge containing 100 g. of TNA, or TNT, was

<sup>92</sup> Z. ges. Schless.-u. Spreng., 1913, 8, 185.

<sup>93</sup> J. Ind. Eng. Chem., 1920, 12, 870.

<sup>94</sup> Waller (*loc. cit.*) says: "The volume of the charge chamber in which explosives are compared in the field is practically a constant, whether it is a borehole or a war head. It would then for even more rational to compare explosives in the laboratory by volume. To do this, the above relative 'strength' figures must be multiplied by 1.86 to 1.90 for TNA, by 1.5 to about 1.75 for the other explosives, and from the 'efficiency' values thus calculated, the 'brisance' maxima are obtained by taking velocities of detonation at these maximum densities."

<sup>95</sup> This refers to the higher rate of detonation of NG (>7000 m./sec.). It has also a lower rate (<2500 m./sec.) (cf. Comey 7th Int. Cong. Appl. Chem., 3, B, 28).

<sup>96</sup> Schweiz. Chem.-Zelt., 1919, 1, 96.

<sup>97</sup> Z. ges. Schless.-u. Spreng., 1918, 11, 112; 1918, 13, 222.

laid crosswise on the bar, midway between the supports, and detonated by a No. 8 cap. TNA tore the iron over a width of  $4\frac{1}{2}$  in. out of 5 in. and folded it completely together. TNT merely bent it to the extent of  $7\frac{1}{2}$  in. in the centre without tearing it.

No. 50. *Comparative brisance of TNA and TNT transmitted through water.* Cartridges composed of varying quantities of TNA and TNT were suspended under water at varying distances from submerged drums made of 1/10 in. steel strengthened by two ribs. In one series of tests, with 1 oz. at 1 ft. distance from a drum  $42\frac{1}{2}$  in. long and  $28\frac{1}{2}$  in. diam., with TNT the indentation produced was 20 in. wide, 13 in. high, and 3 in. greatest depth, but no rib was crushed and a hole was blown on the near side only. With TNA the indentation was  $26\times 21\frac{1}{2}\times 5\frac{1}{2}$  in., one rib was crushed, and holes were blown on both near and far sides.

No. 51. *Comparative brisance in bombs and grenades.* Mills grenades containing 1 oz. each of TNA and other service explosives (tetryl, TNT, etc.) were placed in a wooden box filled with sawdust and suspended from the ceiling of a wooden shed, and detonated to obtain comparative fragmentation. With TNA alone the experiment miscarried, as the floor of the shed was blown through.

No. 52. *Brisance tested on steel plates  $7\times 120\times 120$  mm., with paper cartridges containing 100 g. of TNA.* With the cartridge standing upright a hole was cut clean through. With the cartridge lying lengthwise across the steel plate (which was supported at both ends), the plate was folded completely together. With the steel plate half protruding over the edge of a steel table, with the cartridge lying on the plate parallel to edge of table, just balanced so that a slight touch would cause it to fall to the ground, the plate was cut completely in two.

Tests such as No. 50 have a direct bearing on the efficiency of charges for depth bombs, mines, and especially torpedoes.<sup>100</sup> In torpedo heads the density is of additional importance,<sup>101</sup> as the volume can only be increased at great expense, also because for equal weights the centre of explosion is brought closer to the object to be destroyed.<sup>102</sup> Wet gun-cotton is in this respect even inferior to TNT.<sup>102</sup> The amatols under water are also stated to be inferior to TNT, no doubt owing to their low rate of detonation.<sup>103</sup>

Brisance as evidenced by tests Nos. 48, 49, 51, and 52 is all-important for surface demolition, and particularly again when weight or volume is limited, e.g., by the carrying capacity of bombing aircraft,<sup>104</sup> by the calibre of guns used against fortifications, or by the feasible weight of grenades thrown against tanks.

The brisance of TNA, combined with its sensitiveness to detonation by primers, can also be utilised to explode other materials, by employing it either as an intermediate detonator, or else as a sensitiser admixed to such materials.

No. 53. *Sensitiveness to detonation by fulminate.* Tests by Storm and Cope,<sup>105</sup> 0.4 g. of each nitro-compound per test:—

	Priming charge.		Minimum wt. of primary charge for complete detonation.	
	Fulminate %	Chlorate %	Slightly hand-tamped partially confined.	Compressed at 200 atm., entirely confined.
TNA	100	0		
0.4g.	90	10	0.4500	0.2000
	80	20	0.3125	0.1700
Tetryl	100	0	0.3125	0.1700
0.4g.	90	10	0.3500	0.2400
	80	20	0.3000	—
Picric acid	100	0	0.2750	—
0.4g.	90	10	0.4000	0.2500
	80	20	0.3750	0.2300
TNT	100	0	0.3750	0.2200
0.4g.	90	10	> 1.0	0.2600
	80	20	> 1.0	0.2500
			> 1.0	0.2400

It is the last column (compression and confinement) which corresponds to practical conditions in detonators and fuses. J. Marshall<sup>106</sup> (of the Du Pont Co.) misquotes the results (for fulminate-chlorate) as tetryl=0.2000, TNT=0.2500, and TNA=0.2000 (which latter should be 0.1700 as actually recorded).

No. 54. The Dynamit-A.-G. Nobel, of Hamburg, record<sup>107</sup> that tetryl and TNA both "when they are compressed to their highest density, retain their property of facility to detonate by means of the initial impulse of fulminate of mercury," and point out that friction under firing strain, which may lead to detonation of the primer in a shell fuse containing an incompletely compressed intermediate charge, is thereby avoided.

No. 55. *Sensitiveness of larger quantities of compressed and confined TNA to fulminate.*

Paper cartridges containing 100 g. of TNA and 1 g. or  $\frac{1}{2}$  g. of fulminate each gave two complete detonations in two tests, while with  $\frac{1}{4}$  g. of fulminate three tests gave one complete detonation and two ignitions.

No. 56. *Sensitiveness of wet TNA to an initial impulse.* Using a No. 6 detonator, 50 g. of TNA with 0.55% moisture gave compression of lead block 16.32 mm.; with 5.79% moisture 16.06 mm.; no compression was observed with 10.06% and 15.06% moisture, the TNA failing to detonate.

The following results were obtained using No. 6 detonator and primer of dry TNA:—

Wt. of dry primer.	TNA.	Moisture, %	Comp. of lead block, mm.
5.0	45	15.06	17.98
1.0	49	15.06	16.44
0.75	49.29	15.06	None
1.0	49	15.90	15.14
1.0	49	16.42	None
5.0	45	16.42	None

No. 57. *Comparative power developed by different explosives when detonated by fulminate alone, and by TNA primed with fulminate:—*

Explosive.	Detonator.	Lead block test, (c.c. enlargement/g.)			Ballistic pendulum test, (Net ft. lb./10g.)		
		1.	2.	3.	1.	2.	3.
Nitro-glycerin explosive (blasting gelatin)	No. 6. Fulminate	50	49.5	49.5	1295	1295	1295
	No. 6. TNA	49	48.5	50	1345	1345	1295
Ammonium nitrate, 90% TNT 10%	No. 6. Fulminate	12	16	14	807	767	847
	No. 6. TNA	21.5	22	21.5	858	847	909

<sup>100</sup> As to the fundamental importance of a high velocity of detonation in torpedo-heads cf., e.g., Pramor, Z. ges. Schless.-u. Spreng., 1917, 12, 233.

<sup>101</sup> Cf. Budoft, Z. ges. Schless.-u. Spreng., 1907, 2, 4; Molinari and Quartieri, *ibid.*, 1913, 8, 405, 482; Bichel, E.P. 8950/1911.

<sup>102</sup> Cf. Molinari and Quartieri, *loc. cit.*; Pramor, *loc. cit.*

<sup>103</sup> Cf. Molinari and Quartieri, *loc. cit.*

<sup>104</sup> For rate of detonation cf. Robertson, J. Chem. Soc., 1921, 19, 27.

<sup>105</sup> TNA bombs used in the war have been described in *Illus. London News* (Dec. 26, 1914) (charge of TNA alone), and by Colver "High Explosives," 1918) (charge of 2 parts of TNT and 1 part of TNA). In either case the violence of the explosion is emphasised.

<sup>106</sup> U.S. Bureau of Mines, Tech. Paper 125, 1916.

<sup>107</sup> J. Ind. Eng. Chem., 1920, 12, 336.

<sup>108</sup> E.P. 2407/1912.

The fulminate detonator contained 1.0 g. of fulminate charge, and the TNA detonator 0.3 g. of fulminate charge, the remainder having been replaced by about half its weight of TNA. Similar results were obtained when No. 7 or No. 8 detonators were used. Thus fulminate, acting through TNA, has a higher initiating effect than a fulminate charge heavier than both of these together when employed alone.

No. 58. Relative power developed by TNT shell charges exploded by TNA- and tetryl-intermediate detonators. The conclusion was reached by the officer in charge that "the action of TNA was not inferior and was even superior to the action of commercial tetryl, which is the regular explosive for detonators of the type mentioned."

As to the sensitising properties of TNA when used as an ingredient in explosive compositions, several tests have already been recorded.<sup>108</sup> Substantial progress has since been made, and a few further tests will now be described.

No. 59. Sensitising and pressure effects of TNA when mixed with TNT.

(a) TNT-TNA composition, cast, then ground (TNT solidif. p. 77° C.). With TNT alone the enlargement with a No. 6 cap in Trauzl block (average of 4 tests) was 228.75 c.c.; TNT with 9% TNA 239.25 c.c.; TNT with 20% TNA 264.75 c.c.

(b) TNT-TNA mixtures, not cast (TNT=m.p. 77° C.). With No. 6 cap (5 tests each), TNT alone gave average 235.0 c.c.; with 10% TNA 246.8 c.c. Using No. 8 cap (3 tests each) TNT 275.0 c.c.; TNT with 10% TNA 290.7 c.c.

(c) TNT-TNA mixtures, not cast (standard pure TNT). Using No. 6 cap TNT alone gave 228 and 190 c.c.; TNT with 5% TNA 208 and 217 c.c. Using No. 8 cap TNT alone gave 237, 250, and 174 (P) c.c.; TNT with 5% TNA gave 256, 255, 245 c.c.

No. 60. Sensitising and pressure effects of TNA when cast with dinitrobenzol (DNB), then ground:—Pure TNT gave an enlargement (Trauzl block) 230 c.c.; TNA 25%, DNB 75%, 221 c.c.; TNA 33%, DNB 67%, 240 c.c.; TNA 40%, DNB 60%, 255 c.c.; TNA 50%, DNB 50%, 277 c.c.

No. 61. Sensitising and brisance effect of TNA when cast with dinitrobenzol in shells.

6-pdr. shells, loaded with a cast composition of 3 parts of dinitrobenzol and 1 part of TNA, were fired against steel plate in same conditions and with identical results as given under No. 44. The following fragmentation was obtained in a 4-inch common projectile:—Weight empty with base and fuze plugs 28.80 lb. Bursting charge 2.7 lb. Mixture "A" (cast) plus 63 g. loose TNA as a booster. Standard 8 dynamite cap and Bickford fuze fitted through fuze plug. Recovered 400 fragments weighing 9.65 lb. Weight of largest fragment 2.16 lb. (base plug), smallest less than 2 grains. Average weight of fragments 0.024 lb. Calculated number of fragments 1161.

No. 62. Sensitising effect of TNA on TNT in detonating fuses. It has already been recorded<sup>109</sup> that, in equal conditions, an admixture of 20% of TNA increases the velocity of detonation of a 2.6-mm. TNT detonating fuse by 12%.

Nitroglycerin (or dinitroglycerol) and TNA appear to be the only materials with which it is commercially<sup>110</sup> possible to sensitise cheap ingredients so as to obtain powerful explosives of high density, good oxygen balance, and a high rate of detonation.

<sup>108</sup> Z. ges. Schless- u. Spreng., 1913, 8, 185.

<sup>109</sup> *loc. cit.*

<sup>110</sup> Tetryl has also been proposed for this purpose, for instance in "Fortex" with ammonium nitrate (Mem. Poudres et Salp., 1913-14, 128), but its brisance is not such as to compensate for its high cost of manufacture.

### No. 63. Types of industrial TNA explosives.

(1) Dry. (a) Substituting TNA for TNT etc. to obtain higher density without loss of sensitiveness and thereby reduced hygroscopicity and higher power. Examples: TNA 35%,<sup>111</sup> ammonium nitrate 65% (compressed to 1.6 sp. gr.); TNA 50%,<sup>111</sup> barium nitrate 40%, potassium nitrate 10% (compressed to 1.8 sp. gr., no packing required). (b) Substituting TNA plus cheap and comparatively inert ingredients for TNT etc. Example: TNA:Di-nitrobenzol (1:3) 12%<sup>111</sup>; ammonium nitrate 88%. (c) Adding TNA to black powder.

(2) Plastic. (a) Substituting TNA plus a liquid or semi-liquid material for NG in dynamites to increase power by reducing amount of inert material necessary for oxygen balance; to achieve greater safety in manufacture, transportation, and use, avoid risks from exudation,<sup>112</sup> volatility,<sup>113</sup> or freezing, and prevent deterioration on storage<sup>114</sup> or through warm climates. Example: TNA 35%, liquid nitrated solvent naphtha (about 14% N) 5%, nitrocellulose 0.2%, combustible matter 2.2%, sodium nitrate 57.6%. The pressure, velocity of detonation, and sensitiveness of this explosive were found to be close to those of a corresponding 40% NG-dynamite. (b) Sensitising cheap plastic compositions by means of TNA, to obtain increased power and reliable detonation<sup>115</sup> at increased densities. Examples: TNA 20%, liquid nitrated solvent naphtha (14% N) 9.7%, nitrocellulose 0.3%, barium nitrate 70%. This gave good detonation with No. 8; it was not tested with No. 6 detonator.

	%				
TNA .. ..	2.5	5	4	2.75	2.5
Dinitrobenzol ..	—	5	—	—	—
Liquid nitrated solvent naphtha ..	6	8	10	6	5
Ammonium nitrate (coated) ..	88.5	82	82	83.75	84
Nitrostarch ..	5	—	—	7.5	6
Starch ..	—	—	2	—	—
Nitrocotton ..	—	—	2	—	—
Consistency ..	powdery	plastic	plastic	plastic	powdery
Detonation with No. 6 detonator	good	good	good	not taken	not taken
Detonation with No. 8 detonator	good	fair	fair	not taken	not taken
Small block compression (No. 7 detonator) (75 g. explosive)	13.18 mm.	not taken	not taken	12.0 mm.	13.12 mm.

Mixtures of TNA, liquid nitrated toluols, and a chlorate or perchlorate in varying proportions may be used according to nature of chlorate selected and degree of nitration of the toluol.

It appears remarkable that the enormous brisance of TNA can be reduced to such an extent as to

<sup>111</sup> Some comparative tests carried out with these explosives have been described in Z. ges. Schless- u. Spreng., 1913, 8, 185.

<sup>112</sup> To which sodium and ammonium nitrate dynamites are particularly liable (cf. Marshall, "Explosives," 2nd ed., p. 363).

<sup>113</sup> Which leads to "the severe and nauseating headaches and other disagreeable and dangerous maladies which result from the handling of explosives containing nitroglycerin" mentioned in U.S.P. 1,309,553 by Swint (du Pont Powder Co.).

<sup>114</sup> Thus gelatinised dynamites suffer on storage in warm climates, blasting gelatin quickly becomes difficult to detonate and loses in power, sodium nitrate gelignite cartridges are particularly liable to remain unexploded after storage, and NG has a tendency to revert to its lower rate of detonation, and large consignments of such explosives have to be condemned from time to time (cf. Marshall, "Explosives," 2nd ed., p. 368; Kast, Z. ges. Schless- u. Spreng., 1913, 8, 89, 175; Stettbacher, *ibid.*, 1913, 13, 405; Hargreaves, J., 1914, 337; Mann and Kirton, Report to Govt. of West Australia, J., 1919, 848A).

<sup>115</sup> Thus potassium and still more sodium chlorate explosives are generally difficult to detonate even at moderate densities; sodium and ammonium nitrate and sodium chlorate explosives give the more trouble through insensitiveness due to hygroscopicity, less compressed they are; plastic nitrostarch (cf. Swint, *loc. cit.*) and nitrated solvent naphtha explosives and other plastic non-NG explosives (cf. Schweizer, Z. ges. Schless- u. Spreng., 1910, 14, 23) generally tend to be too insensitive, and TNT as a sensitiser is insufficiently active and cannot be used in sufficient proportions on account of its great oxygen deficiency (cf. U.S.P. 1,357,768, Cook and Harris, Atlas Powder Co.).

permit of large proportions of the same being embodied in propellants for fire-arms.

No. 64. The following is an example of a composition which gave the required velocity *within the permissible pressure*, when fired with a 150-g. bullet in a O'3 Service rifle:—

60 parts of finely ground TNA was embodied with 40 parts of soluble nitrocellulose by means of 92 parts (by weight) of pure ethyl acetate,<sup>114</sup> and worked into powder of 0'0602 in. diam. by 0'2185 in. length.

Charge.	Initial velocity.	Pressure.
42 grains	ft. per sec.	lb. per sq. in.
42 5 ..	2523 ..	48,750
43 5 ..	2593 ..	47,400
44 ..	2647 ..	52,100

The weight, and still more the volume, of such a charge are substantially less than for a corresponding nitrocellulose powder charge. These tests have only been preliminary, and it is not yet possible to state either the maximum velocity obtainable within the permissible pressure, by further changes of composition and dimensions of the powder, or the nature of stabilisers, if any, which may possibly be required.

#### VI.—NOTES ON THE MANUFACTURE OF TNA.

Experience has shown the nitration of *m*-nitraniline to TNA to be an exceedingly safe operation, and it has all along been carried out as an ordinary chemical—not an explosive—manufacture. This is due to the great solubility of *m*-nitraniline in sulphuric acid, which excludes any formation of separate layers, such as may occur with mixed acid and toluene or nitrotoluenes; also to the high melting point of TNA, which causes it to be obtained as a finely divided, crystalline suspension in the spent acid (which cannot be detonated even after filtration), and thus prevents—unlike TNT—the formation of separate layers of explosive and spent acid; perhaps also to the relatively low temperature of nitration, though according to tests on an experimental plant scale the temperature may be allowed to rise uncontrolled without danger of explosion; and, lastly, to the fact that the nitration is a single-stage operation, and can therefore easily be made fool-proof.

The nitration has hitherto been carried out in units up to about 2500 American gallons capacity. The control has been found so easy that there is no objection to the use of even larger units. The time occupied by a nitration has been reduced to about 6 hours. After separation from the undiluted spent acid by filtration the TNA is water-washed and again filtered. It is then ready for drying. With 20% of water content it cannot be detonated by any method (*cf.* No. 56), and is admitted on the U.S. railroads as a non-explosive.

The drying of TNA corresponds to the drying of picric acid, *i.e.*, it is relatively safe. Several methods have been employed, but drying on shelves, by means of hot air fans, has given the best results.

The latest operating results indicate the following net consumption of materials, per lb. of TNA produced, including all intermediate stages (dinitrobenzol and *m*-nitraniline): Benzene 0'6 lb., sulphuric acid (100% basis, including 5% reconcentration loss) 2'3, nitric acid (100%) 1'0, sodium sulphide (60%) 1'0, sulphur (lumps) 0'4 to 0'5 lb. About 17 lb. of sodium thiosulphate can be recovered, but has hitherto been run to waste. The above sulphuric acid consumption can be substantially reduced where the high—and superfluous—heat test requirements for military TNA are waived, also where the reconcentration loss of the relatively large amounts of sulphuric acid used is avoided by mixing

the spent acid directly with fresh acid for nitric stills. This has been found possible in actual practice, since the spent acid retains neither explosive nor volatile organic matter, and since it is recovered undiluted.

Starting from a reasonably pure *m*-nitraniline, the yield previously given for the final stage (130–140 parts of TNA per 100 parts of *m*-nitraniline)<sup>115</sup> has been confirmed on plant scale.<sup>116</sup>

#### CONCLUSIONS.

In 1913, on the basis of results obtained on an experimental scale, it was stated<sup>117</sup> that TNA possesses valuable properties in a high degree; that it is a well-characterised crystalline chemical compound, neutral, without action on metals; that it is the strongest solid explosive,<sup>118</sup> has a higher density than any other purely organic explosive, is non-hygroscopic, does not change on storage, is specially resistant to heat and friction, not over-sensitive to mechanical shock, can be easily detonated by an ordinary detonator even when highly compressed, has a smaller oxygen deficiency than other aromatic explosives, that it is a very suitable ingredient of explosive mixtures, and can be made at a competitive price.

All of these conclusions have been confirmed by later developments on an industrial scale. In addition, it has been proved that TNA is not injurious to health, and that it does not change through moisture, during manufacture, storage, or use—such change requiring more or less prolonged exposure to a combination of moisture and temperature which both substantially exceed the normal, whereas in practice, where temperature may exceed the normal (in shells, bombs, etc.), moisture does not, and where moisture may be high (in submarine mines etc.), the temperature is not.

The successful development of the industrial production of TNA has been achieved largely by the skill and perseverance of American technical men and operators.

Fleet, Hampshire.

#### THE DETERMINATION OF ZINC BY THE POTASSIUM FERROCYANIDE METHOD.

BY E. OLIVIER.

The determination of the zinc content of Australian concentrates is usually carried out volumetrically, either by the so-called American method (the potassium ferrocyanide method) or by the Schaffner method (titration of the ammoniacal

<sup>114</sup> Z. ges. Schiess- u. Spreng., 1913, 8, 185.

<sup>115</sup> Van Duin and Van Lennep (Rec. Trav. Chim., 1917, 37, 114) on the basis of a few laboratory nitrations of aniline and of *m*-nitraniline with potassium nitrate—which are but two of the numerous methods of obtaining TNA described in the author's patents—record somewhat lower yields. In view of the definite examples previously given of nitration temperatures of 100° C. down to below the ordinary (*cf.* E.P. 3224 (1910); Z. ges. Schiess- u. Spreng., 1913, 8, 185), Van Duin and Van Lennep's remark (*loc. cit.*, *cf.* Rintoul, Soc. Chem. Ind. Ann. Repts., 1919, 530) that the author's nitration temperature of 70° C. was too high, is irrelevant; whereas Stettbacher's converse allegation (Z. ges. Schiess- u. Spreng., 1916, 11, 112) that no nitration occurs below 100° C. is entirely incorrect. Subsequently Stettbacher himself (Schiess- und Sprengstoffe, 1919, 201-2) quotes "Van Duin's (*etc.*) method" of nitration at 50° C. as the best. As to the nitration of *m*-nitroacetanilide, attention had already been called (Flügelhelm, G.P. 243,070) to an intermediate compound characterised by its pale yellow needles which had been obtained by Witt and Witte (Ber., 1908, 41, 3091) when using two equivalents of potassium nitrate and operating in certain conditions, which they found on analysis to be a new isomeric picramide. Van Duin and Van Lennep (Rec. Trav. Chim., 1920, 39, 145), departing in material respects from the indications given by Witt and Witte, were thereby misled into erroneous suppositions which will be dealt with elsewhere, in connexion with a discussion of the purely scientific aspect of the mechanism of these nitrations.

<sup>116</sup> Z. ges. Schiess- u. Spreng., 1913, 8, 185.

<sup>117</sup> It can now be added that it has a higher shattering power than any other explosive.

<sup>118</sup> This solvent was selected as it does not react with TNA, whereas alcohol does.

solution with sodium sulphide); this last method is also sometimes termed the Vieille Montagne method (*cf.* Fiévet, *Bull. Soc. Chim. Belg.*, 1919, 23, 351).

In the American method, as usually carried out, the ore or concentrate is decomposed by means of nitric acid and potassium chlorate, the mixture evaporated to dryness, the residue boiled with ammonia and ammonium chloride solution, and the filtrate acidified; the copper is removed by means of lead foil, the liquid is neutralised with ammonia, a solution of potassium hydrogen tartate and ferric chloride is added, the solution heated to about 75° C., and titrated with standard potassium ferrocyanide solution in presence of ammonia until a spot test with acetic acid shows a blue coloration.

The method used for preparing the solution for analysis results in the extraction of the zinc together with the copper, cadmium, calcium, and magnesium, and part of the lead, iron, and manganese. Of these elements only the zinc, copper, cadmium, and manganese are precipitated by the ferrocyanide, and only the copper is removed prior to the titration, so that the cadmium and manganese present are returned as zinc. Whilst the amount of cadmium present in the concentrates is usually so small as to be negligible (it averages about 0.1%), this is not so in the case of manganese, which may be present to the extent of considerably more than 1% and so result in a high figure being returned for zinc. In one case, for example, the author found 1.16% of manganese (as  $Mn_2O_3$ ) in the zinc solution before titration.

That amounts of manganese such as are found in concentrates vitiate the titration is shown by experiments in which manganese corresponding to 2% and 4% respectively of  $Mn_2O_3$  was added to solutions of known zinc content; on titration of these solutions 0.8% and 2.3% or zinc in excess of the quantity present was indicated.

To separate the manganese completely from the solution, a few c.c. of hydrogen peroxide are added to the mixture of ammonium chloride solution and ammonia used to treat the residue resulting from the decomposition of the ore; this renders the manganese insoluble, and the results obtained agree closely with those found by the Schaffner method.

When the percentage of manganese present is small (*e.g.*, 0.25%) the results obtained by the two methods (without the use of hydrogen peroxide) agree within about 0.1%, but in most cases manganese will be present in amounts sufficiently great to necessitate the use of hydrogen peroxide.

The American method has another disadvantage, especially where a large number of titrations have to be made daily—*viz.*, the titration is carried out in hot solutions; this is necessary in order to obtain complete precipitation of the zinc ferrocyanide in a dense, gelatinous form. Further, the blue coloration produced in the spot test is not entirely satisfactory, since although the reaction is very sensitive the colour is not always very distinct in presence of coloured ferrocyanides and the intensity of the colour is not appreciably increased with increasing amounts of potassium ferrocyanide.

In the author's opinion, the Schaffner method is preferable to the American method in view of the above facts, and also having in mind the fact that the zinc is precipitated as colourless sulphide, which affords a ready indication of its freedom from other metallic sulphides. The only other colourless insoluble sulphide known is the double sulphide of zinc and cadmium, discovered by the author in 1886, but the error arising from this source is negligible since, as already pointed out, zinc ores contain very little cadmium, and moreover the greater part of the cadmium is separated as sulphide together with the lead and copper.

Central Laboratory of the Vieille Montagne.



## Glasgow Section.

Meeting held at Glasgow on April 22, 1921.

MR. J. H. YOUNG IN THE CHAIR.

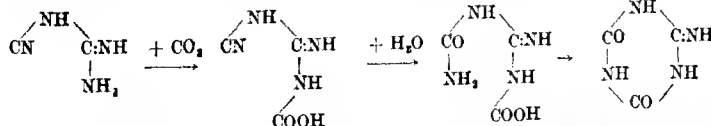
## THE PREPARATION OF GUANIDINE SALTS AND OF NITROGUANIDINE.

BY THOMAS EWAN, M.Sc., Ph.D., F.I.C., AND  
JOHN H. YOUNG, M.Sc., F.I.C.

The methods described in this paper for the preparation of guanidine salts and nitroguanidine easily and economically were worked out between 1903 and 1905. At that time the authors thought they saw a possibility of utilising guanidine nitrate and nitroguanidine industrially.

The possibility of preparing guanidine salts from cyanamide was first investigated. Erlemeyer (Annalen, 1868, 146, 253) obtained guanidine from cyanamide and ammonium chloride in alcoholic solution at 100° C. Volhard (J. prakt. Chem., 1874, [2], 9, 15) heated aqueous cyanamide and ammonium thiocyanate at 100° C. (10 hrs.) and obtained some guanidine thiocyanate. In these papers no mention of yield is made.

The authors first investigated the formation of guanidine nitrate from sodium cyanamide ( $\text{Na}_2\text{CN}_2$ ), a sample containing 72.8%  $\text{Na}_2\text{CN}_2$  being used; 5 g. of this was dissolved in 200 c.c. of cold water and concentrated nitric acid added drop by drop as the cyanamide was dissolving so as to keep the solution as nearly neutral as possible, and finally made just acid to methyl orange with one drop of N/1 nitric acid. The solution, containing cyanamide and sodium nitrate, was evaporated on the water-bath to a small bulk and transferred to a large test tube; 5 g. of solid ammonium nitrate was added and the whole heated in a sulphuric acid bath to 145°–150° C., and kept at this temperature for about one hour, during which time the liquid evaporated considerably and became pasty, but there was no appreciable disappearance of cyanamide. The temperature was then slightly raised and a little more (1 g.) ammonium nitrate added. At 155° C. the mass began to froth considerably and cyanamide slowly disappeared, and after a further  $\frac{1}{2}$  hr. none was left. The product was dissolved in hot water, filtered from a little insoluble matter, and the guanidine determined as picrate. The yield was 43.1% of theory.



Vozarik's method (Z. angew. Chem., 1902, 15, 670; J., 1902, 1041) was employed to determine guanidine quantitatively. A quantity of the substance containing about 0.25 g. of guanidine compound is dissolved in 25 c.c. of water (filtered if necessary), precipitated with 75–125 c.c. of 0.8% ammonium picrate solution (made by dissolving 3.75 g. of picric acid in water, adding ammonia till alkaline and making up to 500 c.c.), and after standing for 1–2 hrs., filtered through a known weight of asbestos in a Gooch crucible, washed twice each time with 12 c.c. of ammonium picrate solution, drained well on the pump and dried at 100° C. to constant weight; 1% of the weight of the precipitate and 2.5% of the weight of asbestos

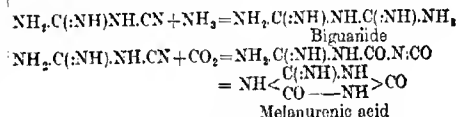
is allowed for ammonium picrate retained, and the solubility of guanidine picrate in ammonium picrate solution is taken as 1 in 4000.

A number of preparations of guanidine nitrate, starting with sodium cyanamide, were made with yields usually near 50% theory. The operation may be divided into the following stages:—(1) Dissolving the sodium cyanamide in dilute nitric acid to form a solution neutral to methyl orange and containing cyanamide and sodium nitrate. (2) Evaporation of this solution to dryness in the water-bath under reduced pressure (50 mm.). (3) Extracting the cyanamide from the residue by digesting with ether. (4) Evaporation of the ethereal solution. (5) Heating the residue with excess of solid ammonium nitrate to 150°–180° C. to form guanidine nitrate.

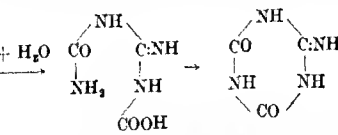
In one experiment the amount of cyanamide at the end of stages (1) to (4) was determined and the quantity of guanidine nitrate at the end of (5). The following were the yields at the respective stages expressed as percentages of the theoretical yield: (1), 100; (2) and (3), 97.9; (4), 95.2; (5), 57.3%. From the above results it appears that the loss of yield takes place in stage (5), so it was decided to polymerise the cyanamide at the end of stage (4) by adding a few drops of ammonia and warming; when this was done the subsequent stage (5) gave an almost theoretical conversion of dicyanodiamide into guanidine nitrate according to the equation  $\text{H}_2\text{C}_2\text{N}_2 + 2\text{NH}_4\text{NO}_3 = 2\text{CN}_2\text{H}_4 + \text{HNO}_3$ .

This led the authors to investigate more fully the formation of guanidine salts from dicyanodiamide.

B. Rathke (Ber., 1895, 18, 3102) found that dicyanodiamide heated with ammonium chloride and water at 150° C. gives considerable quantities of guanidine hydrochloride; ammonia and carbon dioxide are also produced and react with dicyanodiamide to form biguanide and melanurenic acid respectively.



G. Bamberger (Ber., 1883, 16, 1074) observed the formation of melanurenic acid from dicyanodiamide and water at 160°–170° C. and later (Ber., 1890, 23, 1856) he formulated the change as follows:—



Smolka and Friedreich (Monatsh., 1888, 9, 1227) obtained biguanide from the product of the interaction of alcoholic dicyanodiamide and ammonium chloride at 105° C. (8 hrs.), and have also (Monatsh., 1889, 10, 86) described experiments in which small quantities of dicyanodiamide and ammonium chloride (1 mol.) or sulphate ( $\frac{1}{2}$  mol.) were heated together in the dry state at 160°–170° C. The products of the reactions were biguanide salts. The melt, when dissolved in water and treated with copper sulphate and sodium hydroxide, gave a rose-red precipitate of biguanide copper sulphate ( $\text{C}_3\text{N}_4\text{H}_8\text{Cu}_2\text{H}_2\text{SO}_4$ ).

Bamberger and Dieckmann (Ber., 1892, 25, 543) say that the formation of biguanide by this method

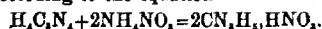
is a very delicate operation, temperature, proportion of reactants, and time affecting the result. They recommend that an intimate mixture, quite dry, of 5 g. of dicyanodiamide and 8 g. of ammonium chloride be heated in an oil bath, with constant stirring, until the temperature reaches 195° C., at which point it is kept for 5–10 mins. and then cooled, extracted with water, filtered from a little insoluble matter (ammelin), and the copper salt precipitated; the yield of copper salt was 45–50% of theory.

Bamberger and Dieckmann also state that Smolka and Friedreich's temperature (160°–170° C.) is too low, and that at 195° C. biguanide decomposes into ammelin. The authors examined Bamberger and Dieckmann's method and concluded that the proportions given (5:8) were due to a printers' error (5:3 is more nearly correct). The following results were obtained:—

Dicyanodiamide, g.	NH <sub>4</sub> Cl, g.	Final temp., °C.	Time to heat up, mins.	Time at top temp., mins.	Copper biguanide sulphate dried at 100° C., g.	Yield % theory.
5	3	190–200	—	6	0.08	0.7
5	3	190–200	—	6½	0.8	7.3
5	3.2	195	—	5½	0.8	7.3
5	3.2	195	—	10	0.42	3.8
5	3.2	185	5	5	1.57	14.3
5	3.2	175	5	5	1.83	17.1
5	3.2	165	2	5	2.00	18.2

It will thus be seen that most of the investigators mentioned above considered a biguanide salt to be the principal product resulting from heating a mixture of dicyanodiamide and an ammonium salt. Where the formation of a guanidine salt was stated no mention of yield was made, and the fact that very high yields can be obtained seems to have been quite overlooked. The authors prepared the following guanidine salts, viz., nitrate, thiocyanate, sulphate, and chloride, by heating mixtures of dicyanodiamide with the respective ammonium salts in the manner described below. In the case of the nitrate and thiocyanate almost theoretical yields were obtained, whilst in the case of the less fusible sulphate and chloride the yields were not quite so high, but nevertheless satisfactory. One of these salts (thiocyanate) was made later in the same way, and a very satisfactory yield obtained by Werner and Bell (Chem. Soc. Trans., 1920, 117, 1133), who found that by heating an intimate mixture of 43.5 g. of dicyanodiamide (95.5%) and 76 g. of ammonium thiocyanate to 120° C. for 3½–4½ hrs. guanidine thiocyanate is formed, and a yield of 90.8% of theory was obtained.

**Guanidine nitrate.**—The following method was found to be satisfactory. The proportions used were according to the equation



A mixture of 300 g. of dicyanodiamide and 570 g. of ammonium nitrate was heated in an enamelled iron pan, 10 in. diameter and 6 in. deep, by means of a Bunsen flame, and stirred with a thermometer. The mass was all fused at 110° C. and was heated to 150° C., at which point the burner was removed as the heat of the reaction kept the temperature up for some time. The mass became more viscous as guanidine formed. As soon as the temperature began to fall the burner was replaced till the temperature reached 180° C., when the reaction usually gives out sufficient heat to increase the temperature of the mass to 180°–190° C., at which point the reaction is soon completed. Finally it was heated to 200° C. for about 10 minutes and poured into iron moulds. 80–95% of guanidine

nitrate was obtained in this way. The time of the complete operation was about 1½ hrs.

Larger hatches were made in the following way:—3 kg. of ordinary commercial ammonium nitrate was melted in an enamelled iron pan 12 in. diam. and 6 in. deep over a Bunsen flame and some 20–30% of the dicyanodiamide theoretically required (say 1.5 kg.) added during the fusion in order to lower the melting point; the remainder was added gradually at 160° C. at such a rate that no external heat was required to keep the temperature steady at this point. When all the dicyanodiamide had been added the mass began to crystallise, so the temperature was raised to 170° C. The mass became liquid again but soon solidified, so the temperature was raised to 180° and finally to 190° C., waiting each time till the mass was pasty. Finally it was heated to 200° C. and poured into moulds. The product contained 91% of guanidine nitrate and the loss of weight was about 3%. 3 hrs. was required for an operation. About 170 lb. of guanidine nitrate was made in this way.

**Guanidine thiocyanate, CN<sub>2</sub>H<sub>4</sub>·HSCN,** was made in a similar way to the nitrate by heating an intimate mixture of 55 parts of dicyanodiamide and 100 parts of ammonium thiocyanate. Yields of over 90% were easily obtained.

**Guanidine sulphate, (CN<sub>2</sub>H<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>SO<sub>4</sub>.** When the ammonium salt is not readily fusible it is more intimately mixed with the whole or a considerable part of the dicyanodiamide before being heated. For example, 100 parts of dry ammonium sulphate is ground together with 60–62 parts of dicyanodiamide (or a considerable part of it) and the mixture fused in an open vessel. The fused mass (to which any dicyanodiamide remaining unmixed is added gradually) is then maintained at a temperature preferably between 150° and 180° C. until it has solidified to a cake of guanidine sulphate. In one experiment 125 g. of an intimate mixture of equal molecules of dicyanodiamide and ammonium sulphate was heated. At 140°–145° C. the product caked together and gave off ammonia; at 157° C. it was semi-fused, and became at 163° C. a thick paste, considerable heat being evolved. At 177° C. it was quite fluid and frothed a good deal, and the temperature was maintained without external heat. The mass was kept at 170°–180° for ½ hour. 111 g. of product was obtained containing 5.67% insoluble in water (ammelin), 1.16% of biguanide sulphate, 93.7% of guanidine sulphate, and a trace of melamine. This represents a yield of guanidine sulphate of 83% of theory.

**Guanidine hydrochloride, CN<sub>2</sub>H<sub>4</sub>·HCl,** was made in a similar way to the sulphate from 100 parts of ammonium chloride and 80 parts of dicyanodiamide.

**Nitroguanidins, CN<sub>2</sub>H<sub>4</sub>·NO<sub>2</sub>,** was prepared by J. Thiele (Annalen, 1892, 270, 1) by treating 100 g. of guanidine nitrate with a cold mixture of 100 c.c. of concentrated sulphuric acid, 50 c.c. of sulphuric acid containing 10% of SO<sub>3</sub>, and 40–50 c.c. of nitric acid (sp. gr. 1.5) and pouring into 2 litres of water after a few minutes. The yield does not seem to be mentioned.

The authors prepared nitroguanidine from guanidine nitrate in three ways, viz., by the action of: (a) sulphuric acid alone, (b) nitric acid alone, (c) a mixture of sulphuric and nitric acids.

**Method (a).**—The solubility of nitroguanidine in dilute sulphuric acid was first determined; 1 g. of powdered nitroguanidine was mixed with a definite quantity of water and concentrated sulphuric acid (sp. gr. 1.84, 82%, 1 c.c. contains 1.69 g. H<sub>2</sub>SO<sub>4</sub> and 0.15 g. water) added, drop by drop, till the nitroguanidine had just dissolved. The temperature of

the saturated solution was 13° C. The following results were obtained:—

% H <sub>2</sub> SO <sub>4</sub> in final acid.	Solubility of nitroguanidine, g. in 100 g. acid.	g. Nitroguanidine dissolved for each g. H <sub>2</sub> SO <sub>4</sub> in the acid.
33.2	2.55	0.077
28.2	1.37	0.042
25.2	0.95	0.038
22.7	0.87	0.038
20.0	0.72	0.036
17.2	0.65	0.037
5.8	0.37	0.064

The sulphuric acid should therefore be diluted so as to make the final acid 20%; 4% of the nitroguanidine should remain in solution for each g. of H<sub>2</sub>SO<sub>4</sub> used per g. of nitroguanidine.

To examine the action of sulphuric acid on guanidine nitrate the materials used were 92% acid as above, and Nordhausen acid (1 c.c. weighs 1.9 g. and contains 62.23% H<sub>2</sub>SO<sub>4</sub> and 33.77% SO<sub>3</sub>). The guanidine nitrate used was crystallised from dilute nitric acid and was colourless and practically pure. Nordhausen acid acts too violently on guanidine nitrate; it also appears to decompose nitroguanidine. In one experiment the nitroguanidine dissolved in concentrated sulphuric acid decomposed suddenly, with evolution of much heat and gas, on adding a little sulphuric anhydride to it.

In the following experiments the temperature never exceeded 30°–40° C.; a little gas is evolved in the warm mixtures—none if the temperature is kept at 12° or 13° C.—but the yield appears to be hardly affected. The mixture was always poured (after standing for a definite time) into sufficient water to dilute the sulphuric acid to 20%. The nitroguanidine which separated was filtered on the pump, washed once with a little cold water, drained well, dried, and weighed.

The action of different quantities and strengths of sulphuric acid on guanidine nitrate, also different times were considered systematically. In experiments using 5 g. of guanidine nitrate and 16.9 g. of acid the highest yields (79.1% in 30 mins., 79.6% in 18 hrs.) were obtained with acid containing 92% of H<sub>2</sub>SO<sub>4</sub>, although almost equally good yields (77.7–78%) were obtained for both times with 87% or 95% acid. Using 5 g. of guanidine nitrate and 25.4 g. of acid for 48 hrs., the highest yield (75.1%) was found with 87.1% acid; with a smaller quantity of acid (8.45 g.) the best yield (84.5% in 48 hrs.) was obtained with 95.5% acid, but yields of over 80% resulted when 92% or 100% acid was used. With 10 g. of guanidine nitrate and 8.45 g. of acid the yields were low in all cases, the best results (36.8% in 22 hrs. and 40.5% in 72 hrs.) being obtained with acid of 102.4% H<sub>2</sub>SO<sub>4</sub>.

To ascertain the effect of temperature on the reaction, 5 g. of guanidine nitrate was dissolved in 10 c.c. of 92% sulphuric acid, without cooling, and placed in a water-bath at 81° C. rising to 84° C. 7 mins. after mixing much gas was being evolved from the acid liquor. The product was poured into water and let stand overnight before filtering etc. Yield 3.03 g.=71.1% theory.

To try whether the nitroguanidine separated completely in a few minutes when poured into water, 5 g. of guanidine nitrate was mixed with 10 c.c. of 92% sulphuric acid, allowed to stand for 25 mins., poured into 65 c.c. of water, allowed to stand for 18 hrs., filtered, washed, dried, etc.; yield 3.35 g.=78.7% theory, which is practically identical with the yield got by filtering at once after pouring into water.

The time allowed for reaction of 5 g. of guanidine nitrate in 5 c.c. of 92% acid appears to influence the yield considerably; e.g., after 10 mins. the yield

was 64.5%; 17½ hrs., 76.5%; 48 hrs., 83.1%. When 5 g. of guanidine nitrate is dissolved in 10 c.c. of 92% acid the time of reaction has little effect; thus after 12 mins. the yield is 78.2%; 25 mins. 78.7%; 30 mins., 79.1%; 3 hrs. 23 mins., 78.2%; 17 hrs. 40 mins., 79.6%.

Method (b).—The solubility of nitroguanidine in nitric acid was determined by dissolving 0.5 g. of nitroguanidine in hot water, cooling to get it finely divided, and adding nitric acid, drop by drop, till the whole was just dissolved in the cold. Results:—

c.c. water.	c.c. conc. HNO <sub>3</sub> .	% HNO <sub>3</sub> in final acid.	g. nitroguanidine dissolved by 1 g. HNO <sub>3</sub> .
15	4.5	27	0.085
25	6.0	23	0.064
40	7.5	19	0.051
50	7.2	15.5	0.053
100	5.3	6.4	0.073

The minimum solubility thus lies at 19% HNO<sub>3</sub>. The concentrated nitric acid used contained 87.6% HNO<sub>3</sub> (1 c.c.=1.3 g. HNO<sub>3</sub> and 0.18 g. H<sub>2</sub>O), and was reddish coloured. 10 c.c. weighed 14.814 g. The following results were obtained using 5 g. of nitroguanidine and nitric acid alone for nitration:—18 hrs.: 3 c.c. of acid, yield=0%; 5 c.c., 11.3%; 10 c.c., 15%; 15 c.c., 49.5%; 96 hrs.: 3 c.c., 0%; 5 c.c., 24.4%; 10 c.c., 53.8%; 15 c.c., 64.5%. The mixtures stood at the ordinary temperature. The 5 g. of guanidine nitrate dissolved completely in 15 c.c., almost completely in 10 c.c., very little in 5 c.c., whilst the 3 c.c. was soaked up completely, leaving a solid mass. The 5 c.c. mixture solidified to a mass of prismatic crystals on standing, and several crystals of the same shape came out of the 10 c.c. lot. Apparently guanidine nitrate is capable of combining with another molecule of nitric acid.

Method (c).—Action of mixture of H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> on guanidine nitrate. The following results were obtained, using 5 g. of guanidine nitrate and allowing to stand for 68 hrs:—

HNO <sub>3</sub> , %	H <sub>2</sub> SO <sub>4</sub> , %	H <sub>2</sub> O, %	Acid mixture, g.	Yield, %
35.1	50.8	14.1	7.4	11.5
50.0	36.1	13.9	10.4	44.4
38.3	60.6	1.1	6.8	65.7
38.3	60.6	1.1	13.6	70.0

The above results show that method (a) is the most satisfactory and that the best yields are obtained by using 5 g. of guanidine nitrate to 5 c.c. of 92%–93% sulphuric acid and standing for 48 hrs., but if time is a consideration very satisfactory yields can be obtained by using 5 g. of guanidine nitrate to 10 c.c. of 87%–95% acid and standing for 30 mins. A batch of about 60 lb. of nitroguanidine was made as follows:—80 lb. of crude guanidine nitrate (90–94%) was dissolved in 147 lb. of 95% sulphuric acid and stirred by hand with an iron rod in a steel ladle, 21 in. diameter and 15 in. deep, set in a wooden tub with cold water flowing. The temperature was kept at 25°–33° C. Six hours was required to get the guanidine nitrate dissolved (this could be reduced by more efficient cooling). The mass was allowed to stand overnight, when it had half solidified; it was diluted with 514 lb. of water in stoneware vessels and the nitroguanidine was filtered on a stoneware vacuum filter. The cake was washed with a little water and then stirred with 100 lb. of water in a stoneware vessel and a portion tested by titrating with N/1 sodium carbonate and methyl orange; 2 lb. of sodium

carbonate was added, which made the liquor slightly alkaline. It was filtered off and drained again, then stirred with a further 100 lb. of water, filtered, pressed well down, and finished with a small wash on the filter. The damp product weighed 94 lb. and contained 43% of water. It was dried in an evaporator till a final product was obtained which weighed 55 lb. and contained 6.57% of water and 0.17% of sodium sulphate. Assuming the guanidine nitrate used to be all 94% (which is too high as it was slightly damp), the yield works out at 80.2% of theory.

This work was done in the laboratories of the Cassel Cyanide Co., Ltd., Glasgow, and our thanks are due to the directors for permission to publish the results.

## London Section.

Meeting held at Burlington House on April 4, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

### THE SCIENTIFIC PRINCIPLES OF COLD STORAGE.

BY PROF. WALTER STILES.

(Abstract.)

From the physico-chemical point of view all foods consist of very complex systems. With the exception of butter, the chief constituent of each system is water in which are dissolved various inorganic salts, while organic substances, particularly proteins and fats, are dispersed through the aqueous phase in the form of an emulsion, or in the colloidal condition.

Most foods are capable of preservation in cold storage, and many systems of classification of such substances are possible according to the point of view adopted. When we have to consider the scientific principles involved, the most satisfactory classification is one based on the temperature of storage. From this point of view we can divide all food materials into two classes. In the first class the food material during storage has to be maintained at a temperature above the freezing point of the food on account of serious deterioration which takes place if the water in the food is allowed to separate out as ice. Fruits are very typical of this group of foods. Many other plant organs, such as tubers and roots, behave similarly. Unless it be in very exceptional circumstances, freezing has so far not been applied with success to the preservation of these, and when kept in cold storage care has to be taken that the temperature does not fall below the freezing point. In the second class much lower temperatures are employed, so that it is ensured that the greater part of the water is frozen. As typical of this group of foods may be cited mutton and rabbits.

These two methods differ greatly with regard to the scientific problems involved. In the first case, the physical condition of the food remains unaltered, but various chemical processes, including enzyme actions, take place, which lead to changes in the composition of the food, which are known as maturing or deterioration according as the changes improve the quality of the food or the reverse. At these temperatures of storage also many foods are liable to be attacked by micro-organisms, which in practically all cases lead to unpleasant changes in the food substance. The changes taking place in foods during cold storage at temperatures above the

freezing point are thus chemical changes that are to be investigated by the methods of organic chemistry, including biochemistry. In the second case, however, in which the food is preserved in the solid frozen condition, we are faced with an entirely different set of problems. The chemical actions which bring about changes in the composition of the food must proceed with extreme slowness, both on account of the low temperature and because the liquid medium in which such reactions take place is now replaced by a solid one. From the freezing point downwards the quantity of liquid becomes less and less until at the eutectic point the whole mass is completely solid. For the same reasons the growth of micro-organisms is either greatly retarded or completely inhibited. On the other hand, freezing involves a change of state of one of the substances of the food, that is, there is a change in physical structure. Cold storage in the frozen state will only be successful if this change in physical structure which takes place on freezing is reversed on thawing, so that in its final state preparatory to use the system comprising the food is nearly identical with the fresh produce. The problems involved here are problems of physical chemistry; the biochemical problems which figure largely in the storage of food above the freezing point take here only a secondary place, but they are not necessarily negligible, especially if storage is continued over a long time.

There are several advantages in keeping food in the frozen rather than in the physically unchanged state. As the result of the much slower rate of the chemical reactions which produce deterioration, material can be preserved for a considerably longer time frozen than unfrozen. Produce can be packed more closely when frozen and, not being nearly so liable to the attacks of micro-organisms, does not require nearly so much attention and inspection.

One of the main objects of scientific research in cold storage should therefore be to transfer as many substances as possible from the class of substances which cannot be preserved frozen to the class of those which can.

As an example of the first class of foods fruit may be selected. The conditions which allow of variation are the temperature and humidity of the air in the storage chamber, illumination, movement and composition of the air in the store, and the possible protection of the fruit by a wrapper of some sort. The stage of development at which it is best to commence the storage of fruit is obviously a matter of importance, and so is the determination of the length of time which the fruit can be kept in store. For the solution of these problems it is clear that a careful study of the changes taking place during the ripening of fruit is necessary. Moreover, there is no reason to suppose that different species of fruit, or even different varieties of the same fruit, go through the same series of changes, so that it appears probable that every species of fruit will have to form the subject of a separate biochemical examination. Such investigations are now being made, and the results should be of the greatest interest for the student of cold storage. The need for such separate investigations is emphasised when it is remembered that the value of each particular species or variety of fruit depends upon something very characteristic in the flavour or aroma.

Since the rate of the reactions involved in ripening of fruit increases with rise of temperature, it may be concluded that, in general, the nearer the temperature is to freezing point, so long as it is above it, the longer can the fruit be stored, and there has been in practice a continuous tendency to use lower and lower temperatures until at the present time temperatures about 0° C. are often employed. As to other conditions in the store, the

degree of movement of the air and the relative humidity are generally determined by the liability of the fruit to lose water by evaporation, or to be attacked by moulds or bacteria. In the latter case air movement with a comparatively dry atmosphere is indicated; in the former, moist still air is more suitable.

What has been said so far will give some idea of the principles involved in the cold storage of fruit; similar considerations will hold for other food substances stored above the freezing point; even a material such as beef, which is usually preserved in this way, is to be considered as subjected to the same scientific principles. It will not be necessary therefore to consider further instances of food maintained above the freezing point, and we may pass on at once to the principles concerned in the preservation of food in the frozen condition.

It has already been noted that the chief difficulties here arise from the change of state from liquid to solid not being reversed on thawing with sufficient completeness. In substances such as rabbits and mutton, which are generally stored in the frozen condition, this reversal is practically complete; in other cases, as in some fish and in beef, with the ordinary methods of freezing the reversal is anything but complete, and such frozen material on thawing yields material inferior in quality both to fresh produce and to that stored for short periods above the freezing point.

We are at once faced with the question of how systems such as meat and fish behave on freezing. Many observations have been made on somewhat similar systems, such as sols and gels of gelatin. If a gelatin gel with a concentration of, say, 2 to 5% is allowed to freeze in a cold room, ice separates out at a number of centres leaving a more concentrated solution of gelatin. As the ice crystals grow there will thus be left a network of gelatin getting more and more concentrated, enclosing in the network solid ice crystals. On thawing the mass, the water resulting is not re-absorbed by the gelatin to any greater extent than gelatin will normally absorb water at a low temperature. A certain proportion of the water will be re-absorbed, but not by any means the whole of it, and so the original system will not be regained after the freezing and thawing processes. If the block of gelatin is squeezed in the hand, water can be squeezed out of the gelatin as readily as it can be squeezed out of a wet sponge.

The same sort of thing happens when vegetable tissues—at any rate, such things as apples and potatoes—are frozen and thawed, while a similar phenomenon is to be noted in the case of beef. When a quarter of beef frozen in air is cut, there is generally a noticeable separation of liquid from the beef ("drip") whereby valuable nutrient substances may be lost from the meat; it is also possible that the alleged toughness of some frozen beef is connected with this. On the other hand, no such difficulty arises with mutton.

Our main problem is, therefore, can we by any means so alter the method of freezing that the separation out of comparatively large crystals of ice can be prevented? Can we so freeze the tissues that the space relations of the water and other constituents are maintained the same in the frozen as in the unfrozen condition? If this be done, we may then hope that on thawing the original system will be renewed.

As a guide in this matter we have the researches of Tammann on supercooled liquids. By varying the degree and rate of cooling it was shown to be possible to vary within wide limits the texture of the resulting solid. By cooling a liquid slowly to a temperature not far below the freezing point there is a high crystallisation velocity but only a few centres of crystallisation form; hence crystals grow rapidly from a few centres, and so a very

coarsely crystalline mass results. Further below the freezing point the number of crystallisation centres increases, so that a finer-grained mass results. Further still below the freezing point the number of crystallisation centres reaches a maximum and at lower temperatures diminishes again until at a certain low temperature the number of crystallisation centres which form in unit time is practically zero. If then the substance is cooled so quickly that it passes rapidly through the temperature interval over which crystallisation centres form at an appreciable rate, the substance should set in a form approximating to an amorphous or glass-like condition as the number of crystallisation centres which form is small, and so is the crystallisation velocity. Working on these principles, Tammann was able to obtain 59 substances out of 153 examined, in the form of a glass, and he expresses the opinion that with sufficiently rapid cooling the majority of substances should be obtainable in this condition.

In the case of a gel or a viscous substance such as the content of the muscle of fish and meat, there is another point of view. The formation of large crystals at a few centres leaving a more concentrated gel forming a network, involves the movement of water molecules through the viscous or jelly-like mass. This movement requires time. For this reason also rapid cooling of such systems as animal juices should prevent the separation of large ice crystals from the general mass.

These considerations suggest that rapid freezing may solve the difficulty of the separation of the constituents of frozen tissues. A number of experiments carried out on the freezing of complex sol and gel systems at different rates supported this conclusion, while there are a number of records in the literature indicating clearly the efficacy of rapid freezing in preserving the relations of the constituents of complex colloidal and organic systems after freezing and subsequent thawing.

The evidence thus seems clearly to indicate the solution of the problem of storing food in the frozen condition. The next step is to discover means for carrying out the freezing sufficiently rapidly. When only small objects are in question this is not as a rule a difficult matter; lowering the temperature may be sufficient, and for small experiments the low temperature of liquid air can be used without much trouble. But the matter is not so simple when we have to deal with large objects, such as large fish and quarters of beef, since the rate of cooling and freezing at any point in the body depends on its nearness to the surface and on the specific heat and thermal conductivity of the body, as well as on the temperature of the cooling medium.

With differences in these respects we need not be concerned here, as most of the food materials with which we have to deal do not differ much in their specific heat and thermal conductivity from water. 75% or upwards of their substance consisting of that compound. But it is of some importance that the thermal conductivity of ice is about four times as great as that of water, while its specific heat is only about one half. On these accounts ice will cool much more quickly than water, other things being equal. Consequently, the passing over of the outer layers of a cooled mass of meat or fish into the frozen state, so far from hindering the further withdrawal of heat from the inner regions, materially assists it.

In order to bring about rapid freezing various devices are possible. The most obvious is to use a lower temperature. But here the cost of maintaining very low temperatures will probably limit the downward extension of the temperature of the cooling medium beyond a certain limit. Strong agitation of the cooling medium will also aid in the more rapid cooling of objects, owing to the breaking

down of temperature gradients. This means can, however, not effect a very great increase in the freezing velocity. A third variable which influences the rate of cooling is the thermal conductivity of the cooling medium. Cooling curves of pieces of beef and other food substances of the same dimensions frozen in a variety of substances have shown without question how greatly the rate of cooling is influenced by the thermal conductivity. We are in practice limited by two considerations, namely, cost and any injurious action of the cooling medium on the food material. Thus mercury, in spite of its high conductivity, is ruled out altogether as a possibility. Of practicable substances water has a fairly high thermal conductivity, but as it freezes at a higher temperature than the food, it is of no use for the purpose. But salt solutions have much the same thermal conductivity as water, and these can be obtained in liquid form right down to their eutectic points. Thus sodium chloride can be obtained in solution down to a temperature of  $-21^{\circ}\text{C}$ ., magnesium chloride down to  $-33.6^{\circ}\text{C}$ ., and calcium chloride down to  $-55^{\circ}\text{C}$ . These temperatures are considerably lower than those generally employed for freezing materials in air—the ordinary method—whereas the thermal conductivity is about 28 times as great as that of air.

By the use, then, of lower temperatures or of a salt solution instead of air as the cooling medium, the rate of freezing can be much increased, and by applying these facts it should probably be possible to achieve the cold storage of any food material in the frozen condition. The use of a cooling medium of high conductivity is, however, more effective in the case of smaller objects than with larger ones. For instance, cylinders of 5% gelatin 1 cm. in diameter in brine at  $-10^{\circ}\text{C}$ . freeze 13 times as quickly as in air at the same temperature, whereas when the diameter is 25 cm. they only freeze 2½ times as fast. For this reason freezing in brine should be more efficient in the case of fish than of large bodies such as hind-quarters of beef. Many experiments with fish on both a small and large scale, in this country and in Holland and Germany, indicate that freezing fish in sodium chloride brine is highly satisfactory with most species of fish, and the method is probably capable of extension to all species of fish. Penetration of salt into the fish is in most cases negligible and on cooking there is no evident salty flavour; in any case the entrance of salt can be prevented by the use of a protective covering if it is thought necessary.

With beef the problem is not yet completely solved. Small pieces of beef, up to about 6 lb. or so, can be frozen in brine so that there is no sign of separation of liquid after thawing. The elimination of "drip" is then only a question of using a suitable freezing velocity. It may be necessary to use a lower temperature than  $-21^{\circ}\text{C}$ ., in which case some solution other than sodium chloride brine would have to be employed. The objection to calcium chloride or magnesium chloride is the unpleasant taste of these salts, but some mutton frozen in calcium chloride brine had not the slightest trace of unpleasant flavour on cooking, and some hindquarters of beef frozen in sodium chloride brine at  $-20^{\circ}\text{C}$ . showed no sign of "drip" after they were thawed, nor of salty taste when cooked. But here a secondary difficulty presents itself. A large body, such as a hind-quarter of beef, has to remain in brine at  $-20^{\circ}\text{C}$ . for from 12 to 24 hours for freezing to be complete to the middle. During this time a considerable quantity of salt enters the surface layers of the tissue, and on thawing the quarter the surface appears to have a dull colour. The meat itself appeared excellent in flavour in the trials made with whole quarters, but the appearance was against it. But the solution of this difficulty should

only be a matter of biochemical research, and one may conclude with confidence that to obtain frozen beef of a quality indistinguishable from that of fresh meat is only a matter of time. Whether it will be economically worth while so to preserve this or any other food material is another matter, which does not concern the scientific investigator as such.

#### DISCUSSION.

The CHAIRMAN said that some 25 years ago considerable interest had been shown in the storage of hops in evacuated metal cylinders. The results were satisfactory and comparable with cold storage; it seemed unfortunate that more had not been done in this apparently promising direction.

Dr. G. SCHACK-SOMMER said that some experiments had been made by Charles Tellier, the pioneer of refrigeration, in which meat and fish were subjected to a vacuum to remove the oxygen from the tissue, before placing them in cold storage. Successful results had also been obtained in preserving soft fruit in cold storage by the use of an atmosphere of carbon dioxide to prevent respiration.

Dr. R. T. COLGATE asked for information as to the best methods of thawing frozen food. It was stated that frozen eggs should be thawed at quite a low temperature.

Mr. E. HATSCHKE said that the freezing and thawing of gelatin gels was not a completely reversible process, and he could not see that the rate of freezing or thawing could ever make it so. The swelling of a gel was also materially affected by the electrolytes present in the water, and particularly by hydrogen and hydroxyl ions. If a gel containing an electrolyte was frozen, the water froze out a good deal of the electrolyte also, and that, it seemed to him, was an additional difficulty in getting the process reversible with animal tissues, which contained electrolytes isotonic with 0.7–0.9% NaCl.

Dr. S. MIALL asked if the author had had occasion to study the conditions under which minute animals could live through freezing.

Mr. C. J. TABOR said that rabbits could not be kept in cold storage for ten months at a temperature of  $8^{\circ}$  or  $10^{\circ}\text{F}$ . without damage. Meat could be kept for 6 or 8 months, and chickens about 6 months. One difficulty in the refrigerated produce trade was the elimination of moulds. His own tests had shown that they were harmless, but it was customary to condemn produce carrying mould growths. A rise of about  $10^{\circ}\text{F}$ . in the temperature of the refrigerating apparatus (say from  $15^{\circ}$  to  $25^{\circ}$ ) was sufficient to start mould growth, although it would not occur if the temperature were kept constant. Once started, a reduction of the temperature did not prevent the growth. The reason for this was that the rise of temperature produced a surface dampness to which the moulds (*Mucor mucedo* and *Penicillium glaucum*) adhered. Another trouble with frozen meats was the yellow discoloration caused by bursting of the fat cells on thawing, whereupon the fat became discoloured and rancid. The freezing of fish always involved loss of flavour; the tissues were more delicate and more readily disrupted by the expansion of their watery contents than in the case of meat. Another form of damage, quite distinct from mould, was "must"; it exhibited no signs of fungoid growth and could not be removed by wiping. Its cause was obscure; beyond a slimy dampness on the goods, and a very disagreeable odour, nothing could be traced; meat so damaged was dangerous to health.

Prof. STILES, replying to a question by the Chairman, said that butter which had been kept in cold storage at  $-10^{\circ}\text{C}$ . for nine months showed no alteration in the fat-soluble vitamins. He was not aware that any experiments had been carried out on the other vitamins. In reply to Dr. Colgate, he



said that German investigators had concluded that if the meat were frozen rapidly enough, so that the space relations were maintained in the frozen condition, it did not matter whether the thawing was rapid or slow. On the other hand, the American, Richardson, advocated slow thawing if freezing had taken place in air, because apparently some of the water that was otherwise lost in the "drip" was re-absorbed if the meat were thawed slowly. All his own experiments had been conducted with the view of trying to get the freezing so rapid that a reversible change on thawing was obtained. He had tried to freeze eggs in brine, but they had always burst. Referring to the freezing of small animals, he said there were references in literature to fish that had been frozen in Lake Superior and lived afterwards, but he did not believe that that fish had ever really been frozen, and he often wondered whether small animals like worms were really frozen. Frogs' muscles, when frozen very rapidly, had been found alive after thawing. Micro-organisms were not always destroyed by freezing; some probably were, but the majority of spores were not, and directly the conditions were suitable for their growth, especially when water condensed on the surface, they would germinate and grow. Fish that had been frozen rapidly in brine was indistinguishable from fresh fish when cooked. Although water expanded on freezing by 10%, this did not burst the tissues. Plant tissue could be frozen, but that did not burst the cells, and the reason why frozen salmon turned rancid was probably connected with the fact that it had been slowly frozen. He doubted if it would turn rancid so soon if it were rapidly frozen.

Meeting held at Burlington House on May 2, 1921.

M<sup>R</sup>. JULIAN L. BAKER IN THE CHAIR.

#### FRACTIONAL DISTILLATION WITH CONTACT RING STILL-HEADS.

BY R. LESSING, PH.D.

In the discussion following the paper on gas scrubbing towers by Donnan and Masson (J., 1920, 239 r) I drew attention to the advantages of packing absorption towers and distilling columns with contact rings of the type described in my British Patent No. 139,880 (J., 1920, 321 a). These rings, as shown in fig. 1, consist of cylinders of approximately equal height and diameter, having a gap in the circumference and a more or less diametrical partition connected on one side with the cylinder, but out of touch on the opposite side. They are disposed indiscriminately in the tower or column at the angles which they find when dropped promiscuously into the vessel. Packing material of a similar kind but consisting of plain cylinders was devised by Raschig (E.P. 6298 of 1914; J., 1914, 907), and has found extensive application for works purposes of various kinds. The provision of the central partition and openings, however, has been found to increase the efficiency of the packing very considerably by adding to the contact surface available in a given tower space, and by enhancing the drainage capacity of the packing.

The further advantage of greater ease in manufacturing these rings in large numbers has made it possible to provide them also for laboratory purposes in smaller sizes than the standard one used in large scale plants, which is usually 1 in. diameter by 1 in. height.

The 1-in. rings are used in a great variety of plants of the chemical and gas industries, amongst which may be cited ammonia scrubbers, oil scrub-

bers, absorbers, washers, tar extractors, carbolate decomposers, carbonating and decarbonating towers, debenzolising stills, distilling columns, and still-heads for many substances such as tar and petroleum products, alcohols and essential oils, also condensers, coolers, and heat exchangers. Indeed they are applicable wherever large interfacial surface for liquid and gas or vapour, combined with a minimum of back pressure, is essential.

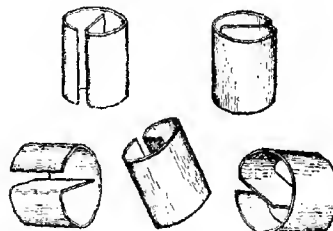


FIG. 1.

The inferiority of laboratory methods to works practice of fractional distillation has been frequently pointed out (e.g. Kreis, *Annalen*, 1884, 224, 259; Silvester, J., 1920, 188 r), and is mainly due to the impossibility of reproducing the factors of time, contact, and distance on the small scale. Since the small rings have been put on the market it has become possible to remedy this deficiency and to use them for the construction of laboratory still-heads on the lines on which the larger ones are applied in manufacturing operations. The early experiments were made mainly with the intention of reproducing as far as possible works conditions in the laboratory, and for this purpose plain tube still-heads as proposed by Hempel were used in which the glass heads were replaced by contact rings  $\frac{1}{2}$  in.  $\times$   $\frac{1}{2}$  in. This made it possible to do away with the principal drawback attaching to the Hempel still-head, viz., choking and consequent "puking" caused by the very considerable resistance offered by the glass beads, which usually take up 80% or more of the still-head space. The resistance of a ring-packed column is so small that still-heads of very considerable height can be used. Incidentally the surface offered by this ring packing is a very large one. It amounts to 8250 sq. cm. per litre (251.5 sq. ft. per cb. ft.), whilst the volume occupied is only about 13% of the total space, leaving 87% of the space free for vapour and condensate.

The advantages of such an arrangement are:—Extreme simplicity—the rings are dropped into plain glass tubes and the use of complicated and fragile products of the glass blower's art is avoided; the same charge of rings can be transferred from one still-head to another whatever its dimensions, or can be used for other purposes in absorption towers or reaction vessels, and are therefore of general applicability; they are not lost if the vessel containing them should get broken.

Apart from these advantages, still-heads packed with these rings have been found to possess a fractionating efficiency which is superior to any known laboratory still-heads or dephlegmators of comparable dimensions. The reason for this must be sought firstly in the large surface per unit of volume which affords ample opportunity for condensation of the vapours and re-evaporation of the condensate; secondly, the turbulence created by the frequent deflection of the currents of vapour and condensate, which can only flow in any one direction for a distance of  $\frac{1}{2}$  in. or less. Thus, formation of "cores" and "channelling" is avoided, and the release of vapour trapped in the condensate is promoted by the continual exposure of fresh surfaces.

A not inconsiderable portion of the action is due to surface tension effects of liquid forming films along the points or lines of contact between adjoining rings. These films or pockets are more or less effective seals, to be broken by the ascending vapour current and thus ensuring intimate contact and interchange between liquid and vapour phase. This amounts to a very considerable bubbling effect which is increased or decreased with the rate of drip of condensate. In order to enhance this bubbling effect and also to keep all surfaces well irrigated, it is desirable to induce as much return-flow as is compatible with the drainage capacity of the column and the avoidance of excessive back-pressure. If the still-head is used unlagged, as is done with the majority of "dephlegmators," it acts largely as a reflux condenser with a rapidly decreasing quantity of condensate towards the top. The bubbling therefore diminishes from bottom to top, and the surface effect of the upper portion of the still-head is not utilised to the full extent. For this reason it is preferable to lag the still-head with asbestos, cotton wool, or corrugated cardboard, thereby avoiding premature condensation, and to provide a reflux condenser which allows the required portion of vapour to pass uncondensed and to distil over, whilst the bulk is condensed and returned, copiously wetting the whole length of the still-head.

This reflux condenser need not be worked as a "regulated temperature" still-head (see F. D. Brown, *Trans. Chem. Soc.*, 1880, 37, 49), but merely serves to re-condense that portion of the vapours which it is desired to use for the irrigation of the still-head and not to send over into the receiver. In this connexion it may not be generally known that the ratio of vapour collected as distillate to that returned into the still can be determined calorimetrically by connecting the water outlet of the final condenser with the inlet of the reflux condenser. The ratio of the rise of temperature in the reflux condenser to that in the final condenser indicates directly the proportion of return flow to distillate. By this mode of working the adjustment of the degree of fractionation for a given height of still-head can be effected, at any rate on the large scale, and may also be occasionally of use in the laboratory.

Where long still-heads are used, a water- or oil-cooled reflux condenser can be dispensed with by leaving a short length of the upper end of the tube unlagged, relying on atmospheric cooling.

A still-head packed with rings may, then, be regarded as a scrubber in which the ascending vapours are scrubbed with the descending condensate. All considerations applied to the theory and practice of scrubbing or absorption towers are therefore applicable to still-heads, and their study should yield valuable results if carried out from the scrubbing point of view.

However well a still-head is lagged there is bound to be a certain amount of radiation and gradual drop of temperature from bottom to top. If a sufficiency of scrubbing liquid is to flush the whole length of the column, radiation must be compensated by the sensible and latent heat of the vapours. In cases where the heating of the still would become excessive, or the quantity of material is insufficient, the loss by radiation may be made up by external heating of the still-head—say by means of electric resistance wires. It is also important to pay attention to the specific heat and conductivity of the packing material.

Whilst the drainage capacity of the packing is very high, the rings of necessity retain a thin film of the liquid after draining. On account of the large surface involved this is more than, for instance, in Young's evaporator still-head, which is

specially designed to retain a minimum of condensate. This point acquires importance only in cases where very small quantities are fractionated and the distillation of the last fraction is desired. In preparative work or in analysis where large enough quantities are available, this drawback disappears entirely as the error becomes negligible, or may be corrected in many cases by steaming the column and recovering the distillate. The amount retained varies according to the viscosity of the substance, its wetting power for various metals, the physical condition of the rings, and the height of column. For instance, aluminium rings retain after 30 minutes' draining 1–2 c.c. of toluene per 100 c.c. of column space, whilst a retention as low as 0.06 c.c. by the same volume was found for a petroleum spirit after some hours' draining.

The construction of these still-heads is exceedingly simple. Tubes from 2.5 to 4.0 cm. wide and from 40 to 150 cm. long will cover the range of most ordinary requirements. The usual constriction at the bottom for connecting through a cork stopper should be as wide as possible to avoid choking with condensate. The tubes may be fitted with a side-tube at the top when no separate reflux condenser is used, or may have an outlet fixed through the top stopper with or without the interposition of a reflux condenser. In glass tubes as a rule no support for the rings is required. They are merely dropped into the tube in a slanting position; on turning it up the rings will arrange themselves and bridge over the bottom shoulder.

If metal tubes are used, as is the case on a semi-manufacturing scale, wire gauze of as large a mesh as will support the rings should be fixed at the base, and practically the whole cross-section of the tube is then available for the discharge of condensate into the still and the upward current of vapours.

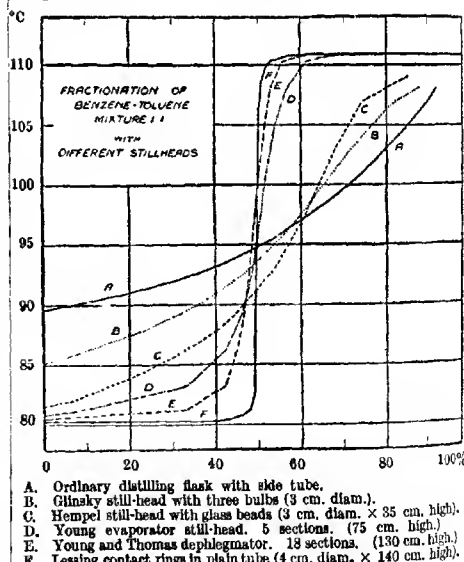


FIG. 2.

In fig. 2 the separating efficiency of a contact ring still-head is compared with that of a number of other still-heads, the classical example of a 1:1 mixture of benzene and toluene being selected. Curves A, B, and C are plotted from values given by Friedrichs (*Z. angew. Chem.*, 1919, 32, 340); D and E are taken from Young's work (*Trans. Chem. Soc.*, 1899, 75, 708). Curve F represents a distillation with a plain

tube still-head 4 cm. wide and 140 cm. high filled with 1-in. aluminium contact rings, at a rate of 60 drops per minute.

The dimensions of the various still-heads are stated on the graph, and although they are not directly comparable, the almost complete separation is very striking. Benzene and toluene distilled over to the extent of 47% and 48% respectively, each within 1°, leaving only 5% of the total mixture in the middle fraction.

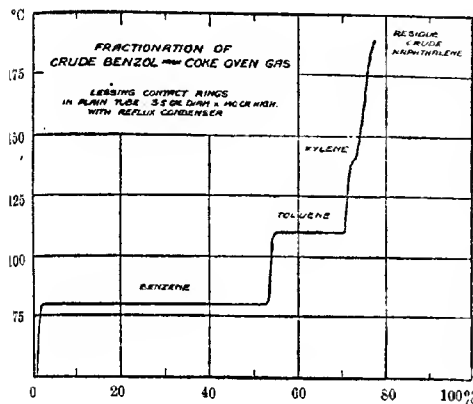


FIG. 3.

Fig. 3 shows the fractionation of a commercial sample of crude benzol obtained from the washing of coke-oven gas with creosote oil. In this case 2000 c.c. was distilled from a metal still in an oil bath with a ring-filled still-head 3.5 cm. diameter by 140 cm. high. A short reflux condenser was employed. These conditions explain the extreme sharpness of the curve, which even indicates the presence of a small quantity of xylene. The sample contained over 20% of naphthalene and wash-oil, and the residue solidified on cooling.

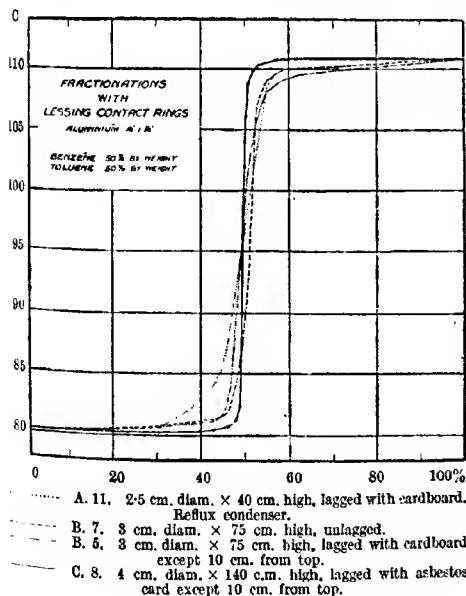
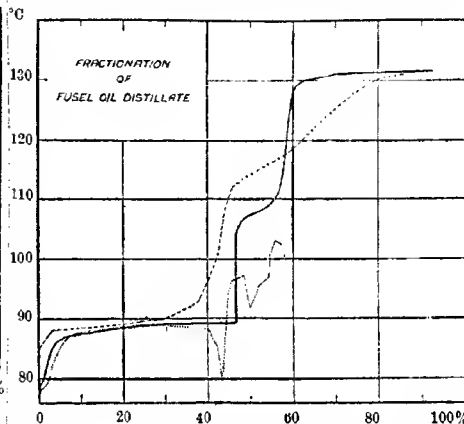


FIG. 4.

A. 11. 2.5 cm. diam. x 40 cm. high, lagged with cardboard. Reflux condenser.  
B. 7. 3 cm. diam. x 75 cm. high, unlagged.  
C. 5. 3 cm. diam. x 75 cm. high, lagged with cardboard except 10 cm. from top.  
C. 8. 4 cm. diam. x 140 cm. high, lagged with asbestos card except 10 cm. from top.

In fig. 4 four distillations are plotted in order to show the influence of the still-head dimensions, heat insulation, and reflux condensing. This graph proves clearly that a certain minimum of scrubbing surface is required in order to obtain the best results. Curve C8 is identical with Curve F in fig. 2.

Very interesting results were obtained in the fractional distillation of a commercial fusel oil distillate of sp. gr. 0.840 at 15°/15° C. The results of



Young 12-pear-bulb column.  
Lessing copper contact rings in plain tube 4 cm. diam. x 140 cm. high.  
Lessing aluminium contact rings in same tube.

FIG. 5.

a preliminary distillation of this sample with a Young 12-pear still-head (fig. 5) show the type of curve obtained by the test customarily employed in many works laboratories.

The following results were obtained on fractionating 1000 c.c. from a glass flask with a glass tube still-head 140 cm. high by 4 cm. wide, filled with 1-in. copper contact rings and lagged with asbestos cord to within 10 cm. of the top. The exposed portion was covered with corrugated paper during the distillation of the fractions above 100° C. Rate of distillation 5 c.c. per minute. At 130.0° the still-head was replaced by a smaller one 40 cm. high by 2.4 cm. wide, and distillation continued at a rate of 7 c.c. per minute. This change accounts for the loss of 1.4%. Volume of distillate was noted for every rise of 0.2° and plotted in fig. 5.

#### Fractionation of fusel oil distillate with Lessing contact-ring still-head.

Fraction no.	Temperature °C. Corr.	% by vol. Total.	% by vol. Fraction.	Sp. gr.	$n_D^{20}$
I.	73.0—87.4	9.5	9.5	0.858	1.3714
II.	87.4—89.6	19.5	10.0	0.870	1.3745
III.	89.6—89.0	24.6	5.1	0.872	1.3750
IV.	89.0—89.5	46.9	Top layer 20.1 Water 2.2	0.861	1.3800
V.	89.5—107.2	50.6	3.7	0.814	1.3900
VI.	107.2—117.0	53.2	7.6	0.808	1.3921
VII.	117.0—130.0	62.3	4.1	0.814	1.4005
VIII.	130.0—131.0	69.8	7.5	0.815	1.4029
IX.	131.0—131.4	92.4	22.6	0.814	1.4034
X.	Steam distillate	95.5	3.1	0.832	—
XI.	Residue in flask	98.6	3.1	0.822	—
Loss	..	—	1.4	—	—

In the preceding table the percentages of fractions as taken off, together with their specific gravities and refractive indices, are recorded.

Fraction I. contains a mixture of aqueous ethyl and *n*-propyl alcohols. The bulk of fractions II. and III. is propyl alcohol. The first drop of free water came over in repeated experiments at 89.1°, and sharply indicates the beginning of the isobutyl alcohol. Fraction IV. was collected in two layers, showing that the mixture of constant boiling-point contains more water than the alcohol is capable of dissolving at room temperature. The rise of temperature, after the last drop of water contained in the sample had distilled over, was so sudden that the curve shows an almost perfect right-angle at this point. The remainder of the isobutyl alcohol came over in anhydrous condition in fraction V., after which amyl alcohol was collected in the remaining fractions.

The distillation of the same sample, carried out under identical conditions except that aluminium rings were used in the place of copper ones, gave somewhat curious results, which seem worth recording. They are plotted in the dotted curve (fig. 5). This curve is, for the first 40% of distillate, practically identical with the copper ring curve, except for a slightly sharper separation in fraction I., probably due to the difference in the specific heat, and therefore rate of warming up of the two metals. The appearance of the first drop of free water at the same point has already been noted. After 40% of the distillate had come over, the temperature being constant at 89.1°, it suddenly dropped. At the lowest point reached, 79.6°, the escape of gas carrying white fumes with acrid smell was noticed. The thermometer then rose again repeatedly, when at 97.2° a similar drop and subsequent rise occurred, which was once more repeated at 103.0°. The distillation was then stopped, as a solid deposit was noticed in the distilling flask. The residue in the flask set solid on cooling, and after steam-distilling the alcohol from flask and column, the solid was found to be voluminous aluminium hydroxide, of which more than 200 g., equivalent to 35 g. of  $Al_2O_3$ , was collected.

The aluminium rings were found to be attacked very considerably, those in the lower layer being pitted and pierced with holes, and a good deal of metal was dissolved away. This observation confirms the results of previous workers (Gladstone and Tribe, *J. Chem. Soc.*, 1876, 29, 158; Seligman and Williams, *J.*, 1918, 1607), who found that the higher alcohols when anhydrous attacked aluminium violently, and that such action is inhibited by traces of moisture.

As an explanation for the abnormal distillation curve it is suggested that at the time when the temperature drop occurred the alcohol in the flask and lower part of the still-head was anhydrous. In reacting with the aluminium, alcohol was used up by the formation of aluminium butoxide to such an extent that the amount of vapour reaching the thermometer was insufficient to keep it heated. The failure of vapours to pass through the still-head caused air to diffuse back, and as the condenser was at that time wetted with a mixture of butyl alcohol and water, traces of the latter on reaching the rings stopped the reaction. Hence, the sudden rise and second drop of temperature when the water was again cleared out from the still-head and when this phenomenon was repeated. The most interesting feature in this connexion from the point of view of fractionation is the fact that when the action occurred, and judging from the experiment with copper rings, the column must have still contained 69 c.c. of the mixture of butyl alcohol and

water of constant boiling point, partly in the form of vapour and partly in the form of condensate. The division between the moist and anhydrous portions of the column was therefore an extremely sharp one, and the rings acted as a perfect seal against moisture. Whilst this experience shows a drawback of aluminium, which in many cases is preferred to other metals, it indicates possibilities of usefulness of ring-packed still-heads which have not yet been exploited.

In conclusion, I wish to express my indebtedness to Professor Sydney Young, F.R.S., for his helpful criticism, to Mr. H. E. Burgess for permission to publish the graph and data of the fusel oil distillation, to Messrs. Barnsley Main Colliery Co., Ltd., for their permission in regard to the crude benzol curve, and to Messrs. C. H. Butcher, A. H. Raine, and F. S. Shadholt for carrying out the experimental work and preparing the drawings.

317, High Holborn, W.C. 1.

#### DISCUSSION.

The CHAIRMAN asked Dr. Lessing if it would be possible by using the apparatus described to increase the strength of the ordinary 93% industrial spirit. Those who in the course of their work used alcohol as a precipitant for dextrans, gums, etc., obtained large volumes of low strength alcohol (80%). It was tedious to use lime for dehydrating such spirit, and it would be a great convenience if the column was applicable in such a case.

Dr. M. W. TRAVERS said that the probability that any particular ring would be so placed that the opening at the side would be exactly at the bottom, which was the only condition under which it would facilitate drainage, appeared to be very small. The same remark applied to the opening between the edge of the feather and the inner wall. Would it not be better to make the rings of perforated metal?

Dr. R. C. FARMER asked if any definite comparisons had been made with the original plain rings and the present ones, which were split. In his experience the split rings tended to link together, and this would presumably interfere with their efficiency.

Dr. F. M. PARKIN said that channeling was very tiresome in most cases, and even in this case surely there was a chance of it taking place. In distillations in reference to benzol and toluol, he had used shell turnings, which was the only material available at the time, and to prevent channeling wooden rings were placed between the turnings, so that the vapour was diverted into the centre again. He found that extremely useful, and it was also useful in scrubbing. Working on the laboratory scale, he had prevented radiation by using a silica tube with pear-shaped condenser and an evacuated outer casing; this on a small scale worked very well, but on a commercial scale it would probably be too expensive.

Mr. CHRISTENSEN asked if rings of non-conducting metal would be preferable to aluminium.

Capt. O. J. GOONWIN said he was not satisfied that it was always necessary to have such a long tube as suggested by Dr. Lessing. He also thought it would be a good plan to decrease the size of the rings from the bottom upwards; that would lessen the surface and reduce the quantity that had to drain back, and the quantity of liquid that was retained on the filling. The size of the rings was really of very great importance, and experience showed that the greatest scrubbing surface was necessary at the top of the column.

Mr. P. PARRISH said that Dr. Lessing had failed to deal with the practical aspect of the question, and there should have been some comparisons between the laboratory experiments and actual practical working. The author appreciated the importance of surface area and local pools as important factors affecting efficient fractionation, but he would find that columns of the character described when applied on the practical scale would be infinitely better if arranged in such a way as to afford actual and positive bubbling. The fractionation of alcohol was still effected on the large scale in Coffey stills, where bubbling was the salient principle.

Dr. H. P. STEVENS asked for information as to the time taken to fractionate the liquid when comparing different distillation heads.

Dr. LESSING, in reply, said that so far the dimensioning of still-heads or scrubbers had been entirely empirical. The graduation of the rings, as suggested by Capt. Goodwin, had already been applied on the large scale. Dilute ethyl alcohol (50–93%) could easily be fractionated to a concentration of 95%; beyond that it became theoretically impossible. With regard to Dr. Travers' point, the majority of rings did get into a position where drainage was facilitated, even if some were lying with the gap on top. The liability of the rings to nest in each other was dependent on efficiency in manufacture, because, according to the thickness of the material, he thought it would be possible to make the slit of such a size that this would not happen. The trouble so far had been that they had not had sufficient control over the raw material; there was sometimes a spring in the material if it were not annealed to the correct hardness, and the rings then opened out more than they were intended to. He had tried the effect of dehydrating agents on alcohol fractions, but they were not of very great use. With any alcohol higher than propyl alcohol, with repeated distillations it was possible to get out practically 50% of the water each time, so that with two or three distillations it was possible to get 70 or 80% more of the alcohol in the anhydrous form without the aid of a dehydrating agent. With regard to the channeling, mentioned by Dr. Perkin, with shell turnings there would be the very difficulty which was avoided with rings, namely, a cork-screw action, which carried the condensate down and allowed the vapour to rise, whilst in the case of the rings it was broken and deflected. Vitreous rings could not be made on the small scale in the thin sections in which they would be required, and on the whole, he was inclined to the opinion that metal rings were preferable on account of their conductivity and quicker exchange of heat between the two phases. He did not agree with Mr. Parrish's remarks; a set of experiments had been carried out during the war on the debenzolisation of benzolised oil in which an ordinary distilling column of the bubbling type had been displaced by a plain gas pipe which had exactly half the cubic capacity of the other still, and was filled with rings; the debenzolisation efficiency rose from 60% to 92 or 93%, and inversely the purity of the mixture of benzol and toluol was increased to a similar degree, so that the benzol-extracting capacity of the return oil was about 90% as against 60–70% in the other case. He had found that channeling was very much more likely to happen with perforated rings than with solid rings; there was a straight run up the tube and dripping right down without the deflection which was obtained with the solid ring. The rate of distillation was very important and was recorded in every case. Generally a higher rate of distillation could be employed when contact rings were used without impairing the efficiency.

## Manchester Section.

Meeting held at Textile Institute on March 4, 1921.

MR. JOHN ALLAN IN THE CHAIR.

### NORMAL AMYLBENZENE AND SOME OF ITS DERIVATIVES.

BY L. GUY RADCLIFFE, M.Sc.TECH., F.I.C., AND N. SIMPKIN, M.Sc.TECH., A.I.C.

The present investigation refers only to normal amylbenzene and its derivatives, as considerable difficulty is experienced in preparing pure iso-amyl compounds, and the ordinary iso-compound is easily converted into a tertiary compound, whilst on the other hand straight-chain compounds can be readily obtained pure.

Normal amylbenzene,  $C_6H_5.CH_2.CH_2.CH_2.CH_2.CH_3$ , was discovered by Schramm (Annalen, 1883, 218, 338); its preparation by the action of sodium on a mixture of benzyl bromide and normal butyl bromide has been described in considerable detail. The hydrocarbon is a colourless liquid with an agreeable odour and boils at  $200.5^\circ$ – $201.5^\circ$  C. under 743 mm. pressure.

In the research to be detailed, Schramm's method of preparation was used with certain modifications, for example, benzyl chloride was used in place of the exceedingly unpleasant benzyl bromide, with good results. The adoption of this method was further influenced by the following considerations:—(1) Normal butyl alcohol can be readily obtained almost pure, whereas amyl compounds from which the required hydrocarbon could be made are extremely difficult to obtain pure. (2) The mechanism of the reaction is fairly simple. (3) It is possible that important derivatives of *n*-amylbenzene may be discovered and thus afford a means of utilising the considerable stocks of *n*-butyl alcohol which are available. In reality the reaction does not correspond exactly to the equation:—



Subsidiary reactions occur in which, e.g., two benzyl radicals unite to give the well-known dibenzyl and further, two butyl radicals couple to form *n*-octane; and it is clear that other reactions must take place, as there is always formed a large quantity of thick oil having a very high boiling point. The effect of these secondary reactions is to reduce the yield of *n*-amylbenzene to about 25% of the theoretical yield.

#### Experimental.

The first preparations of *n*-amylbenzene were made by treating a mixture of benzyl bromide and normal butyl bromide with sodium, the conditions being modified in various ways with a view to increase the yield of amylbenzene and decrease the amount of by-products; with this same object in view benzyl chloride was substituted for the benzyl bromide. The effect produced by diluting the reacting mixture with benzene was also exhaustively studied.

The investigation of the *n*-amylbenzene itself was, after determining its physical properties, mainly confined to a partial study of its sulphonation, in which was included the preparation and properties of a monosulphonic acid and its barium salt, also the corresponding sulphonic chloride and sulphonamide and a phenol. The constitution of the sulphonic acid has been determined, whilst the mononitro compound has been prepared and its properties investigated.

In order to establish the constitution of *n*-amylbenzene with certainty, *n*-amyl bromide was synthesised from *n*-butyl alcohol and coupled with bromobenzene in order to produce amylbenzene by a very different method. A comparison of this amylbenzene with that made by Schramm's method showed that they were identical. The *n*-butyl bromide used in the experiments was made by treating a mixture of commercial butyl alcohol (sp. gr. 0.8133 at 15.5° C., b.p. 115°–118° C.) and red phosphorus with bromine. Using 212 g. of the alcohol, 36 g. of phosphorus, and 255 g. of bromine, a yield of 87.7% was obtained. Many experiments were carried out in order to ascertain the conditions under which the best yield could be obtained.

The benzyl bromide was made by the direct bromination of boiling toluene, and the yield of pure product was about 60%, obtained in the form of a colourless liquid, b.p. 198°–202° C. This product kept well and did not appear to undergo decomposition even after standing for several months.

The benzyl chloride used in the experiments was obtained by the fractionation of the commercial product, the portion boiling from 176° to 179° C. being used for the experiments. It appears worth recording that much uncertainty exists as to the true boiling point of benzyl chloride.

The *n*-amylbenzene was prepared as follows, using 128 g. of benzyl bromide, 103 g. of *n*-butyl bromide, and 34.5 g. of metallic sodium. Sodium in the form of wire was added gradually to the mixed benzyl and butyl bromides; the temperature of the mixture soon rose to 35° C., whereupon it was cooled by immersing the vessel in water. After about one-fourth of the sodium had been added, the temperature being kept below 50° C., the action appeared to slacken and the mixture was heated to about 80° C. Fresh quantities of the sodium wire were added, and when about half the sodium had been added the mixture was allowed to stand overnight. The contents of the vessel now consisted of a solid layer (sodium bromide) surmounted by a pale yellow liquid. As the large amount of sodium bromide present apparently hindered the reaction, the mixture was diluted by the addition of 100 c.c. of dry benzene and gently boiled on the water-bath. The remainder of the sodium was gradually added, and the boiling continued for a further 25 hours to complete the reaction. At this stage the liquid was dark yellow in colour and possessed a pleasant odour, while the whole of the sodium had been converted into the bromide. The mixture was heated by means of an oil-bath, and about 60 c.c. distilled off. The residue was then steam-distilled to obtain the amylbenzene, which collected on the surface of the water as a bright, colourless liquid. Soon crystals began to appear in the distillate, whereupon the receiver was changed and steam distillation continued for another hour, making in all a total of 9 hours. The residue in the distillation flask consisted of the sodium bromide solution, upon the surface of which floated a thick, dark yellow oil. The white, needle-like crystals melted at 52° C. and were dibenzyl. The thick oil has not yet been investigated.

The colourless oil resulting from the steam distillation was carefully fractionated, and gave a 27.6% yield of crude *n*-amylbenzene, b.p. 195°–205° C. Further quantities of dibenzyl were also obtained. The yield is not good, and the secondary reactions resulting in the formation of dibenzyl, octane, and the high-boiling thick oil, are not at present clear.

It was found not to be advisable to dilute the reacting mixture with benzene at the beginning of the experiment. In the other preparations of amylbenzene, benzyl chloride was used with butyl bromide both with and without dilution with benzene, and it was found that the benzyl chloride gave

just as good a yield as the benzyl bromide. As a result of many experiments, in which the conditions were slightly varied, it was concluded that the following conditions tend to give the best yield of amylbenzene:—

1. The sodium wire (sodium cut into slices was also used, but was slower in its action, especially towards the end of the reaction, unless the shavings were very thin) should be added slowly to the mixture of benzyl chloride and butyl bromide, the latter being in slight excess of the theoretical amount required, and the mixture shaken or frequently stirred.

2. The reacting mixture should not be diluted with benzene, and should be kept as cool as possible, i.e., compatible with the continuance of the reaction.

3. To complete the reaction, it is advisable to let the whole mixture stand for a few days with an occasional shaking, since the application of heat, or the development of heat during the reaction, lowers the yield of amylbenzene and increases the amount of the higher boiling hydrocarbons. A diluent with a boiling point lower than that of benzene, such as pure dry ether (free from any alcohol), might give better results, as the mixture would boil at a considerably lower temperature.

#### Purification of crude amylbenzene.

The fraction boiling at 195°–205° invariably contained traces of the benzyl halide, and to remove this, the crude hydrocarbon was mixed with alcoholic potash and gently boiled in an oil-bath for 9 hours, after which the excess of alcohol was distilled off and the residue distilled with superheated steam. The upper layer of distillate was heated to 105° in contact with thin shavings of metallic sodium for 7 hours. The product was free from halogen. The still impure amylbenzene was then fractionated, using a 4-bulb column, and the product boiling between 200.5° and 202° C., under 761.2 mm. pressure, collected as pure amylbenzene. From careful observation the boiling point would appear to lie between 201° and 202° C. at 761.2 mm., agreeing with the figures given by Schramm, viz., 200.5°–201.5° C. under 743 mm.

#### Properties of *n*-amylbenzene.

Normal amylbenzene is a colourless liquid possessing a rather pleasant odour. It is readily soluble in alcohol, benzene, ether, carbon bisulphide, methylal, chloroform, carbon tetrachloride, and glacial acetic acid. If sulphuric acid is added to the hydrocarbon, drop by drop, a slight brownish sediment forms upon the addition of each drop, and with an excess of acid, or on warming, the amylbenzene dissolves (*cf. infra*). The hydrocarbon does not dissolve in cold 70% nitric acid, but does so on warming the mixture; on cooling, however, the amylbenzene comes out of solution. Further details of the nitration are given later.

The density was found to be:—at 15.5°/15.5° = 0.8664; at 15.5°/4° = 0.8651; at 22°/22° = 0.8624, and at 22°/4° = 0.8602. Schramm gave the density of his amylbenzene as 0.8602 at 22° C., but does not state whether this is compared with water at the same or some other temperature (*loc. cit.*). A quantity of amylbenzene was distilled, and the refractive index of each 5 c.c. of distillate determined by means of the Pulfrich instrument; the index in each case was  $n_D^{20} = 1.4751$ .

#### Sulphonation of *n*-amylbenzene.

After a number of preliminary experiments had been performed, 10 g. of the hydrocarbon was sulphonated by the gradual addition of an equal volume of concentrated sulphuric acid, with frequent shaking. A brownish-white substance was produced with the addition of each drop of acid.



and a slight development of heat occurred. After all the acid had been added the resulting mixture was a brownish-white liquid containing globules in suspension, presumably of unchanged amylbenzene, as the characteristic odour still persisted. To complete the reaction the contents of the flask were heated on the water-bath with frequent shaking for 2 hours, and finally for  $\frac{1}{2}$  hour over a small flame. The liquid, now dark brown in colour, was poured into about 250 c.c. of distilled water and neutralised with barium carbonate, followed by boiling for  $\frac{1}{2}$  hour and filtering. The filtrate rapidly deposited fine white, shining, needle-like crystals which appeared flaky when dry, and were very soft in texture; the residue, consisting of barium sulphate and carbonate, was repeatedly extracted with boiling water, and from each extract small crops of crystals were obtained upon evaporation. Each crop was analysed to determine whether mixtures of the mono- and disulphonic acids were formed under the above conditions. The theoretical percentage of barium in barium amylbenzenemonosulphonate,  $(C_{11}H_{23}SO_3)_2Ba$ , is 23.21%; by analysis, 23.26% was found. Further experiments led to a 54% yield of the monosulphonic acid being obtained; the barium salt crystallised without water of crystallisation.

*Preparation of the sulphonic chloride and sulphonamide of *n*-amylbenzene.*

To determine whether the barium amylbenzenemonosulphonate was a single isomer or a mixture of two isomers, a portion of each crop of crystals of the barium salt was converted into the sulphonic chloride and thence to the corresponding sulphonamide.

The sulphonic chloride was prepared by heating a mixture of the finely-powdered barium amylbenzenesulphonate with powdered phosphorus pentachloride on a water-bath for two hours, with occasional shaking, adding the mixture to water, and extracting the oil with ether. On evaporation the ethereal solution yielded a pale brown oil which solidified to flat, needle-shaped crystals of the sulphonic chloride; this melts at  $38^{\circ}$ – $39^{\circ}$  C., it has a peculiar unpleasant odour, and has the property of remaining liquid at the ordinary temperature for a considerable time. It is readily soluble in ether, petroleum spirit, chloroform, benzene, carbon tetrachloride, and carbon bisulphide, but is only sparingly soluble in cold alcohol.

The sulphonamide was prepared from each batch of sulphonic chloride by heating with powdered ammonium carbonate for about 2 hours, grinding the product with several quantities of cold water, and crystallising the pale brown substance thus obtained from dilute alcohol, from which the sulphonamide separated in white, hair-like, glistening crystals somewhat resembling glass wool. Every batch of crystals melted at the same temperature viz.,  $86^{\circ}$ – $87^{\circ}$  C. It would thus appear that only one isomer was present in the barium salt, as obtained by the above method.

The sulphonamide was analysed by a modification of the Kjeldahl method, and was found to contain 6.18% of nitrogen. Calculated for  $C_{11}H_{23}(C_6H_5)_2N \cdot SO_3NH_2$ , N = 6.17%.

In order to determine the orientation of the sulphonic acid and its derivatives, the amylbenzenesulphonamide was oxidised by boiling with an alkaline solution of potassium permanganate (Pinner, Ber., 1888, 21, 243), and the sulphaminebenzoic acid isolated in the usual way and recrystallised from boiling water; it was obtained in small, flat, needle-shaped crystals, almost colourless, and melting at  $278^{\circ}$ – $280^{\circ}$  C. with decomposition. This observed melting point agrees with that of *p*-sulphaminebenzoic acid, whence it may be concluded that the acid formed by the sulphonation of

*n*-amylbenzene as described above is *n*-amylbenzene-*p*-sulphonic acid, and further, that no appreciable quantity of any other isomer is formed.

The free sulphonic acid itself was obtained from the barium salt in the form of very hygroscopic, needle-shaped crystals, which were exceedingly soluble in water, and further, the potassium, silver, and calcium salts have been prepared.

*n*-Amylphenol.

About 5 g. of potassium amylbenzene-*p*-sulphonate was mixed with 14 g. of powdered caustic potash and transferred to an iron tube, sealed at one end. The powder was washed down with 2 c.c. of water and the mixture stirred. The temperature was gradually raised to  $330^{\circ}$ – $340^{\circ}$  C. and kept thereat for 1 hour, whilst a slow stream of hydrogen was passed into the top of the tube throughout the experiment to prevent oxidation of the phenol. After cooling, the dirty, greenish mass obtained was dissolved in about 40 c.c. of water and the solution acidified, whereupon a thick, pale brown oil separated on the surface. This oil was extracted with ether and distilled, yielding a colourless fraction, boiling at  $262^{\circ}$  C. and giving reactions corresponding to a phenol; it is very sparingly soluble in boiling water, readily soluble in caustic soda, ether, acetic acid, or alcohol; the alcoholic solution gave an olive-green coloration on the addition of ferric chloride, which was discharged by hydrochloric acid. When chilled in a freezing mixture, the amylphenol formed white crystals which melted at the ordinary temperature. It is not considered that this compound was obtained in a chemically pure condition. The benzoylamylphenol, made in the usual manner, crystallised from alcohol in white needles, m.p.  $52^{\circ}$  C. A bromo-derivative was also prepared by adding bromine to the amylphenol dissolved in glacial acetic acid and pouring into water; the product, after repeated crystallisations from dilute alcohol, was obtained in colourless crystals, m.p.  $88^{\circ}$ – $89^{\circ}$  C.

*Nitration of the amylbenzene.*

Attempts to nitrate amylbenzene with a mixed acid composed of sulphuric acid (sp. gr. 1.84) and nitric acid (sp. gr. 1.42) did not give very satisfactory results, as it appeared that nitration was not, in any experiment, complete. Mononitroamylbenzene was, however, obtained by the following method:—40 g. of fuming nitric acid (sp. gr. 1.508) mixed with 20 g. of glacial acetic acid was added slowly to 20 g. of amylbenzene dissolved in a further 20 g. of glacial acetic acid, the temperature being kept below  $20^{\circ}$  C. by external cooling; the mixture gradually turned brown in colour, and, after the addition of all the nitric acid, which occupied about 1 hour, was allowed to stand, when it separated into two layers. When the whole was heated for  $\frac{1}{2}$  hr. in a bath of water at  $50^{\circ}$  C., brown fumes were evolved and a homogeneous liquid was produced. After standing for 12 hours, the heating was resumed for a further hour, the mixture cooled and poured into water containing lumps of ice, whereupon a yellow, oily liquid was thrown down. The mixture was made alkaline with caustic soda solution and extracted with ether. The ether solution, after drying over anhydrous sodium sulphate, was distilled, leaving a yellow liquid having a characteristic odour, the bulk of which distilled under 14 mm. pressure at  $155^{\circ}$ – $159^{\circ}$  C. A determination of nitrogen gave a slightly high result, indicating possible contamination with a small amount of the dinitro-compound. Thus prepared, mononitro-*n*-amylbenzene is a pale yellow oil of sp. gr. 1.0650 at  $15^{\circ}$  C. and miscible with the usual organic solvents. When oxidised by the process used for the sulphonamide, *p*-nitrobenzoic acid, m.p.  $236^{\circ}$ – $237^{\circ}$  C., was

obtained; it was concluded that the nitro-compound was *p*-nitroamylbenzene.

At this stage the preparation of *n*-amylbenzene by a quite different method was undertaken in order to be certain that the compound hitherto studied was in reality *n*-amylbenzene. The process chosen was to treat a mixture of monobromobenzene and synthetically prepared *n*-amyl bromide with metallic sodium, when the following reaction would take place:  $\text{C}_6\text{H}_5\text{Br} + 2\text{Na} + \text{BrC}_4\text{H}_9 = 2\text{NaBr} + \text{C}_6\text{H}_5\text{C}_4\text{H}_9$ . For this reaction *n*-amyl alcohol was synthesised from *n*-butyl alcohol according to the method of Adams and Marvel (J. Amer. Chem. Soc., 1920, 42, 311–315). This, briefly described, entails the conversion of *n*-butyl alcohol to the nitrile *via* the intermediate stage of the butyl bromide, then the hydrolysis and esterification of the nitrile by means of 95% ethyl alcohol and strong sulphuric acid to give ethyl valerate, and finally the reduction of the latter by means of sodium and absolute ethyl alcohol to *n*-amyl alcohol. This synthetically prepared *n*-amyl alcohol was then converted into its bromide by means of the phosphorus halide method, and the resulting *n*-amyl bromide coupled with bromobenzene, using metallic sodium as usual. It is necessary to use absolute alcohol when reducing the ester with sodium, as otherwise the reaction fails. All the materials were highly purified at every stage. The final coupling of bromobenzene and *n*-amyl bromide with metallic sodium gave a product which was identical with that originally prepared by the interaction of benzyl chloride and butyl bromide. The outstanding feature of this last method of preparation is the fact that the yield of *n*-amylbenzene is between two and three times as great as in the previous experiments; it would thus appear easier to introduce an aliphatic side chain into a benzene ring than to add another group to an already existing side chain.

In concluding this paper, it may be mentioned that a highly-odorous aldehyde has also been obtained, but the work has been interrupted, and a description of this product cannot be given at present.

#### DISCUSSION.

Captain F. S. SENNATT said that *n*-amylbenzene would probably be converted into an  $\alpha$ -methyl derivative of tetrahydronaphthalene with considerable ease; such a compound would have a very high boiling point and a considerable percentage might be present in the residual oil obtained by the authors, which might yield on examination some interesting compounds.

Mr. H. G. SHATWELL said if the ring were chlorinated and the end of the chain were also chlorinated, and the resulting compound treated with sodium, it might be possible to cause that ring to close as suggested. Had any attempt been made to reduce the nitro-compound described?

Mr. N. SIMPKIN, in reply, said that a large amount of work had already been done in America on the utilisation of butyl alcohol. Apparently the oil might contain such a substance as benzylamylbenzene. In reply to Mr. Shatwell, certain chlorine derivatives of this hydrocarbon had been prepared, but it had been found that instead of the end of the chain being chlorinated, a mixture of compounds resulted. Furthermore, in order to produce a naphthalene ring as Mr. Shatwell suggested, the side chain would require to be chlorinated at the  $\delta$ -carbon atom, not the  $\epsilon$ - or end carbon atom, in order to couple up with the benzene ring. The compound had been brominated, and several distinct compounds with 1, 2, and 3 atoms of bromine obtained, but not in the pure state. The nitro-compound had not been reduced owing to lack of time.

## Newcastle Section.

Meeting held on March 23, 1921.

PROF. P. PHILLIPS BEDSON IN THE CHAIR.

### THE EFFECT OF SOME PHYSICAL CONDITIONS ON CALCIUM SULPHATE CEMENTS.

BY C. L. HADDON.

Anhydrous calcium sulphate cement bears a number of different names, such as flooring plaster, "Estrich gips," and dead-burnt plaster. A slightly modified form is called Keene's cement.

The actual setting time of flooring plaster depends on the temperature and duration of heating used in its manufacture, but subsequent conditions have the same comparative effect on different samples of plaster.

It was long ago shown that the setting of flooring plaster is due to the anhydrous salt dissolving and being precipitated as crystals of the less soluble dihydrate. These crystals in dilute solutions form radiating bundles of needles, and it was assumed that this occurred in its use as a cement, and that fracture of the cement was caused by the breaking of the interlocking crystals. It was also shown by using a dilatometer that in setting there was first a contraction followed by an expansion. This was explained by Davies as being due to the formation of orthorhombic crystals spontaneously changing to monoclinic crystals. Data as to its superficial expansion—the more important from the practical point of view—are lacking. According to Lunge's "Technical Methods of Chemical Analysis," "increase in volume is not a property of dead-burnt plaster." On the other hand, other authorities state that it expands on setting, but no figures are given. The apparatus used for measuring expansion was Le Chatelier's, as laid down in the Engineering Standards' specification for Portland cement. The dimensions of this instrument are so designed that for relatively small expansions (less than 1%) the measured expansion in centimetres is approximately equal to the percentage linear expansion. It is not a suitable instrument for measuring expansion during the plasticity stage of setting.

The same flooring plaster was used throughout these experiments. Three parts by weight of plaster was mixed with one part of water or solution and placed in the cylinder, and the expansion was measured at different times. The subjoined table shows that:—

(1) The expansion was least when accelerating solutions were used, and greatest when retarders were used.

(2) Wet set plaster, using a 5% solution of a sulphate as accelerator, although reaching its maximum strength within 24 hours, yet continued to expand during recrystallisation.

(3) Wetness has a great effect on tensile strength. With plaster kept weakened in this way, a very much greater expansion resulted. The degree of wetness affects expansion.

(4) The expansion under normal conditions is very small—about 0.1%—and ceases within seven days, i.e., the time for complete setting or drying.

In the case of calcium chloride the expansion was measured after the first day; a further expansion of 0.7% took place when thoroughly re-wetted due to drying before complete hydration.

No attempt was made to measure expansion while the cement was still plastic.

The cause of the expansion is obvious—it is due to the pushing apart of the mass by crystals as they

are formed. An expansion of 5% can easily be produced by using a retarder, and keeping the cement wet.

In dry room.	% Expansion from time of loss of plasticity.			
	1 day.	4 days.	7 days.	14 days.
Mixed with water..	0.08	0.12	0.17	—
" 5% soln. CaCl <sub>2</sub> ..	—	0.40	0.75	0.97
" 5% soln. K <sub>2</sub> SO <sub>4</sub> ..	0.06	0.07	0.08	No further increase.
" 5% soln. FeSO <sub>4</sub> ..	0.06	0.07	0.08	—
Mixed with water placed in water after 1½ hrs.	1.0	1.5	1.85	—

The amount of expansion has an effect on the tensile strength. Briquettes which have expanded 5% have less than a quarter of the strength of similarly made briquettes allowed to dry in air, when the expansion is very much less. Similarly, in test-pieces where the plaster was placed in water six hours after making the briquette, and the briquette broken after seven days when still wet, the tensile strength averaged 200 lb. per sq. in., while when the briquette was allowed to dry in air and placed in water only for the seventh day, the average tensile strength was 250 lb. per sq. in.

In a recent paper (J., 1920, 165—168 r) the author mentioned the weakening of flooring plaster by wetness, and it was suggested that it was due to the lubricating action of water. In order to investigate this, test-pieces were placed in water after six hours, taken out after 14 days, and then left in a warm, dry room till they ceased to lose weight. Briquettes were immersed in different liquids for 24 hours and then broken.

The average tensile strength of the dry briquettes was 470 lb. per sq. in.; after immersion in paraffin it was 470 lb.; in thin lubricating oil, 470 lb.; in methylated spirit, 470 lb.; in a mixture of 2 pts. of spirit to 1 of water, 320 lb.; in a mixture of 1 part of spirit to 2 of water, 250 lb.; in water, 200 lb.; and in concentrated calcium sulphate solution, 200 lb. The test with thin lubricating oil gave the same result at the end of 10 days; figures identical with those of the 24-hr. tests were also obtained with water (10 mins.) and concentrated calcium sulphate solution (1 hour).

Thus mineral oils have no effect on strength, while with mixtures of alcohol and water the loss in strength is dependent on their proportions. Water reduces the tensile strength by one half, as does a concentrated solution of calcium sulphate. Leaving in water for ten minutes, provided that the whole cross-section of the cement is wetted by capillary action, has the same effect, showing that it is not due to solubility of the set cement in water. The small amount of water necessary is shown by the fact that if one end of the test-piece is placed in water, the same weakening effect is produced by the water creeping up through capillarity. In the same way crushing strengths are reduced from about 2 tons per square inch to half that amount.

There is no accepted explanation of lubrication. It seems curious that oil should not act as a lubricant, while water does. Oil certainly acts as a lubricant when one slab of set flooring plaster is rubbed over another. In metals, the crystals are supposed to be covered with some amorphous inter-crystalline cement which is stronger than the crystals, which break in cases of fracture. It is possible that some such amorphous calcium sulphate dihydrate exists, and this is affected by free water. Our present knowledge of physical phenomena is not sufficient to justify a full explanation.

The cementing power of calcium sulphate cement may be described as follows:—Crystals of the di-

hydrate grow in the form of needles, until there is great frictional resistance to disruption and also interlocking. Breaking is due largely to this frictional resistance being overcome, and not to the breaking of the crystals themselves, as is the case with metals.

The value of all published data on the mechanical strength of flooring plaster is impaired by the fact that no standard has been laid down as to the condition of the briquette on breaking. The popular method seems to be to keep the briquettes in a more or less damp room during setting. The effect of different amounts of free water would considerably vitiate the results. The fairest way is to do precisely as is done with Portland cement—leave the briquettes in the moulds for 24 hours, take them out and place them in water, and then break the wet briquettes after a week; this would give comparable results as regards the effect of expansion, and eliminate error as regards wetness. The proportion of flooring plaster to water by weight should be 3 to 1, as this gives a plastic mass of uniform composition, while deviations from these proportions give cements of different strengths.

## Communications.

### A MODIFICATION IN THE COMPOSITION OF IODINE SOLUTION FOR THE REICH TEST.

BY H. M. LOWE, M.Sc.

Standard iodine solution used for testing gases for sulphur dioxide may be conveniently made by substituting for the potassium iodide in which the iodine is dissolved a proportion of caustic soda, insufficient to react with the whole of the iodine, but sufficient to form enough sodium iodide to dissolve the remaining iodine. The formula recommended is: Iodine, 127 g.; caustic soda, 30 g.; water, to 10 litres. Thus the 30 g. of caustic soda replaces 150—180 g. of potassium iodide. The iodine reacts with this to give a solution of iodide and iodate and is quantitatively released as free iodine on acidification with a large or small excess of acid. The sulphur dioxide in the gas it is desired to analyse acts in this way in N/10 solution without further addition of free acid. When the solution is used in great dilution, e.g., N/100 to N/1000, as in treating exit gases, it is necessary to add some acid previous to use.

Experiments with this solution showed that the theoretical amount of iodine is always liberated on acidification, whether with a large or small excess of acid. Titration with standard thiosulphate of the iodine thus liberated forms a convenient method of standardising the solution for use. It is immaterial whether the acid is added all at once to the iodine or in small portions as the titration proceeds, but the latter method avoids the iodine being thrown out of solution at any time.

If desired the solution may be titrated with free SO<sub>2</sub> without addition of acid.

Comparison tests on the inlet gases to a Grillo oleum plant showed identical results with the ordinary solution or with the modified solution.

When the solution is diluted to 10 times its bulk it forms a reagent for testing the exit gases from the converter. In this case it is necessary to add to the solution to be tested a small amount of sulphuric or hydrochloric acid together with the starch indicator, as at the dilution used (about N/1000) a very appreciable concentration of acid is required before the iodide and iodate react instantaneously. At a dilution of N/2000 and a bulk of 200 c.c., on the addition of 6 c.c. of N/10 H<sub>2</sub>SO<sub>4</sub> to a neutral mixture of iodide and iodate, a blue colour develops

slowly; with 7 c.c., however, the colour is obtained instantaneously. In the test, therefore, the addition of 1 c.c. of 10%  $H_2SO_4$  is ample to ensure the instantaneous working of the indicator.

The sulphur trioxide present, together with the dioxide at the exit of the converter, would be thought to provide the necessary acidity, and, indeed, usually does so; but on one or two occasions the test failed, and the blue colour was discharged prematurely; it was found, however, that the addition of a little acid caused the blue colour to be reformed. It is therefore advisable always to add a little acid to the test solution before commencing.

The  $N/100$  solution may be standardised against thiosulphate in the same way as the  $N/10$  solution and titrated with sulphur dioxide solution after acidification. Tested on exit gas from a Grillo converter the  $N/100$  solution gave results identical with those using the ordinary potassium iodide solution.

The  $N/10$  solution is sufficiently stable for ordinary purposes. Thus, a 10-litre bottle kept full showed no change in strength after a month, and one half full showed no change in strength after eight days.

H.M. Factory, Gretna.

#### THE ESTIMATION OF RESIN ACIDS IN FATTY MIXTURES.

BY DAVID McNICOLL, M.A., B.Sc.

Twitchell's method for the estimation of resin acids in fatty mixtures (J., 1891, 1894), though it was a distinct advance on the methods then known, has many faults, as pointed out by Lewkowitsch (J., 1893, 502). Since that time no improvement has been made as regards accuracy accompanied by simplicity. The method detailed in this paper not only eliminates the faults of Twitchell's method but simplifies it by the substitution of methyl for ethyl alcohol and of naphthalene- $\beta$ -sulphonic acid for gaseous hydrogen chloride.

It is well known that with Twitchell's method, resin acids undergo decomposition with the production of water-soluble acids of lower molecular weight, and it is the formation of these in varying amount which causes the irregular results. Lewkowitsch has also shown that esterification of the fatty acids is far from being complete.

**Volumetric method.**—About 2 g. of the mixture is dissolved in 20 c.c. of a 4% solution of naphthalene- $\beta$ -sulphonic acid in dry methyl alcohol and heated under a reflux condenser for 30 mins. with the addition of several pieces of porous plate to ensure regular ebullition. A blank experiment with 20 c.c. of the sulphonic acid solution is also carried out for the same time, as the titration value increases slightly during the process. The best results are obtained by heating the flasks on an electric plate or asbestos wire gauze or in an oil-bath; esterification is not so complete when a water-bath is used. The contents of both flasks are then cooled and titrated with  $N/2$  methyl alcoholic potassium hydroxide solution. For unknown mixtures the resin acids are calculated on the combining weight of 346, as suggested by Lewkowitsch.

**Gravimetric method.**—The neutralised solution obtained above is employed. The contents of the flask are transferred to a separating funnel—the volume being noted—and the flask washed out with an equal volume of water made slightly alkaline with potassium hydroxide. After shaking, the mixture is extracted with an equal volume of a mixture of ether and petroleum spirit, the flask having again been washed with the solvent. Separation is instantaneous. The small amount of insoluble soap that usually separates is drawn off with the soap solution, which is extracted twice in similar fashion. The united extracts are washed with a little 50% aqueous alcohol and the washings

added to the resin soap solution, which is then acidified and extracted twice with ether.

**Esterification of fatty acids.**—Pure fatty acids are completely esterified by the method described, as shown by the following figures:—

		c.c. $N/2$ KOH used.	
		Experimental.	Blank.
Cotton oil fatty acids, 2 g.	..	8.50	8.50
Oleine, 2 g.	..	8.52	8.50
Palm kernel acid oil, 2 g.	..	8.57	8.50

The distilled olein, ruby red in colour, showed by titration after treatment the presence of 0.14% of free fatty acids calculated as oleic acid. With the palm kernel "acid oil" an acidity of 0.51% as oleic acid was found. The residual acidity in these two cases is doubtless due to colouring matter which, while being of an acid nature, is not esterified.\*

Lewkowitsch has shown (*loc. cit.*) that Twitchell's method leaves 2–6% of fatty acids uncombined.

**Treatment of resin.**—On submitting resin to the process, partial esterification takes place amounting to 1–3% of the resin acids present; the amount varies for different qualities of resin.

A number of mixtures of known composition were examined by the methods described; the errors in the volumetric method varied between -0.22 and -0.56% and in the gravimetric method between -0.38 and +0.35%. The results obtained by Twitchell's method (as quoted by Lewkowitsch) show errors varying between -9.73 and +5.26%.

The following results were obtained from three different samples. In each experimental figure the value of the blank test has been deducted:—

Resin, g.	Before treatment. c.c. $N/2$ KOH.	After treatment. c.c. $N/2$ KOH.	Resin found. %
1.0473	6.00	5.93	98.8
2.4295	14.32	14.02	97.9
1.2159	7.08	6.90	97.4

That esterification had occurred was confirmed by saponification, 1.2 c.c. of  $N/2$  potash being absorbed in the first experiment. The difference from the esterification value can be accounted for by oxidation of the alcohol.

That there is no appreciable production of water-soluble-decomposition products was proved in the first experiment by the recovery of 1.0375 g. of resin, which absorbed 5.88 c.c. of  $N/2$  potash after evaporating the resin soap solution, dissolving in water, and extraction with ether after acidification.

#### Examination of mixtures of known composition.

Details of the examination of three mixtures by volumetric and gravimetric analysis are recorded below; in the experimental figures the value for the blank has been deducted.

Mixture.		Volumetric method.				Gravimetric Resin. % Found.
		Titration. c.c. <i>N</i> /2 KOH.		Resin. %		
Fatty acid.	Resin.	Calc.	Found.	Calc.	Found.	
2.0391	0.2652	1.53	1.57	11.51	11.84	11.73
1.4320	0.6091	3.46	3.52	28.84	30.28	30.23
1.0624	1.0030	5.73	5.70	48.60	48.45	48.88

\* The amounts of resin found are corrected for the 5% unsaponifiable matter present.

Research Laboratory,  
British Oil and Cake Mills, Ltd., Hnll.

\* Further experiment has substantiated this assumption. The estimation of true fatty matter in the by-products from the refining of cotton oil will be dealt with in a later paper.

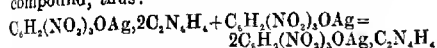
## A DIRECT METHOD FOR THE DETERMINATION OF DICYANODIAMIDE.

BY ERLING B. JOHNSON.

R. N. Harger (J. Ind. Eng. Chem., 1920, 12, 1107; J., 1921, 92A) has recently described a gravimetric process for the determination of dicyanodiamide (cyanoguanidine) in cyanamide and mixed fertilisers, based on the precipitation of a compound of silver picrate and 1 mol. of dicyanodiamide,  $C_2H_4(NO_2)_2 \cdot OAg, C_2H_4N_2$ .

Some years ago the present author, working with Mr. Berhom, isolated the same complexes as those described by Harger in the attempt to obtain a method for the determination of dicyanodiamide, but as it was important to have as rapid a method as possible, a volumetric process was worked out.

It is practically impossible to determine dicyanodiamide volumetrically by means of the above compound on account of the large excess of silver solution which must be used. On the other hand, it was found that under certain conditions the compound of silver picrate with 2 mols. of dicyanodiamide was so insoluble and changed so slowly into the monocompound, thus:—



that a volumetric method could be based on the determination of the amount of standard silver solution used to effect the precipitation. It is necessary to use weak solutions, a low temperature, and excess of picric acid.

The method is carried out as follows: When the sample being analysed contains 5–15% of dicyanodiamide nitrogen 5 g. is used, or a correspondingly larger amount if less nitrogen is present. The sample is transferred to a 500 c.c. flask and 450 c.c. of cold water added. If the sample is calcium cyanamide or other product containing lime, glacial acetic acid is added to bring all the lime into solution as acetate. By this means the nitrogenous compounds present are dissolved much more rapidly and completely than without any acid. A small excess of acetic acid does not matter. Stronger mineral acids isomerise the nitrogen compounds. The solution obtained with acetic acid is suitable for a Kjeldahl determination of the total water-soluble nitrogen, but this is not possible if nitric acid has been used, as has been suggested, to shorten the period of shaking.

The flask is shaken in a machine for 3 hours, the solution is made up to 500 c.c. and filtered; 5 c.c. of 20% nitric acid is added to 100 c.c. of the filtrate, followed by 20 c.c. of sodium picrate solution heated to 40° C. (7.5 g. of picric acid neutralised with caustic soda and diluted to 100 c.c.), and the solution is cooled to about 5° C., and titrated with N/22.4 silver nitrate solution, which is added drop by drop with constant shaking. About 2 c.c. more silver solution is used than is required by the amount of dicyanodiamide believed to be present. The dicyanodiamide compound is precipitated in a more or less gelatinous form according to the amount of dicyanodiamide present. After vigorous shaking the mixture is stood aside for fifteen minutes at 5° C., during which time it is agitated two or three times, then diluted to 200 c.c. with cold water, shaken and filtered. The excess of silver solution used is estimated by titration with N/22.4 sodium thiocyanate in 100 c.c. of the filtrate after adding a further 5 c.c. of 20% nitric acid and about 2 c.c. of 5% ferrio sulphate solution as indicator.

If 5 g. of the sample has been treated according to the method described, every c.c. of silver solution used corresponds to 1% of nitrogen as dicyanodiamide.

To obtain good results some corrections must be applied, but each observer should work these out for himself, as they will depend largely on the personal equation.

The corrections as worked out by the author are shown in the following tables.

*Influence of the excess of silver solution.*—As the compound of silver picrate with 2 mols. of dicyanodiamide changes into the compound with 1 mol. in presence of excess of silver salt, it is possible that such excess may influence the results. This is shown in the following table. Pure dicyanodiamide was used with the addition of 1 c.c. of 20% nitric acid.

Dicyanodiamide-N.	Excess of silver solution.				
	2 c.c.	3 c.c.	4 c.c.	5 c.c.	6 c.c.
0.05 g.=5%	98	103.6	103	105.3	108
0.10 g.=10%	99.5	99.7	100	100.9	101.5
0.15 g.=15%	97	97	97.5	98	98.7

*Note.*—In this and the succeeding tables the figures given are the percentages of the amount actually present.

*Influence of increasing addition of nitric acid.*

Dicyanodiamide-N.	Excess of 20% nitric acid.		
	1 c.c.	5 c.c.	10 c.c.
0.05 g.=5%	98	94.8	90
0.10 g.=10%	99.5	97.4	95
0.15 g.=15%	97	90.4	85.5

*Influence of nitric acid and different amounts of calcium acetate* (the latter would be present in analysing samples of calcium cyanamide or other products containing lime).

Dicyanodiamide nitrogen.	Additions.						
	Nitric acid.	1 c.c.	1 c.c.	1 c.c.	5 c.c.	5 c.c.	10 c.c.
	Calcium acetate.		1 g.	5 g.	1 g.	5 g.	5 g.
0.05 g. = 5%	—	93	90	80	102	82.4	84
0.10 g. = 10%	—	99.5	98	89	99.8	92.4	95
0.15 g. = 15%	—	97	98.7	94.7	97.5	95.3	90

*Influence of temperature on the precipitation.*—2 c.c. excess of silver solution was used in each case.

Dicy. N present.	5° C.	10° C.	15° C.	20° C.	25° C.
0.10 g.=10%	99.6	98	96.4	90.5	85.1
0.05 g.=5%	98	95.5	92	85.5	70

It will be obvious that when working with calcium cyanamide the use of 5 c.c. of 20% nitric acid per 1 g. of calcium acetate present and the addition of 2 c.c. excess of silver solution will give good results without any corrections.

A characteristic feature of the precipitation of dicyanodiamide with a greater excess of silver solution and picric acid is the change of the gelatinous di-compound first precipitated into the small crystals of mono-compound; this occurs more or less rapidly according to the excess of silver and the concentration.

The method has been tested in the presence of urea and dicyanodiamidine (guanlylurea), but neither of these compounds was found to influence the results.

The following table shows the reliability of the method and gives the results of the analyses of very old samples of calcium cyanamide.

When chlorides and soluble sulphides are present a blank test must be carried out by titrating the silver solution without addition of picric acid.

form it does not dissolve completely in amyl alcohol. In such cases the urea must be liberated.

It is important that the reagents and samples used should be as free from water as possible.

North-Western Cyanamide Co.,  
Odda, Norway.

Sample.	New method (picrate).				Old method (modified Caro).			
	Cyanamide nitrogen.	Dicyanodiamide nitrogen.	Urea nitrogen.	Total.	Cyanamide nitrogen.	Dicyanodiamide nitrogen.	Urea nitrogen.	Total.
1	1.11	10.67	1.14	12.92	1.11	10.62	1.21	12.94
2	0.86	10.16	1.06	12.08	0.86	9.64	1.69	12.60
3	0.86	10.81	1.27	12.94	0.86	10.22	1.29	12.37
4	0.77	10.49	1.59	12.85	0.77	10.61	1.62	12.90
5	1.12	10.44	0.94	12.52	1.12	10.46	1.00	12.58
6	13.25	1.76	0.45	15.46	13.25	1.80	0.4	15.45

Further research showed that the formation of dicyanodiamide-silver complexes is not limited to picric acid. The reaction is typical of all nitrophenols which are soluble in water. Compounds of the silver salts of dinitrophenol, trinitroresorcinol, and trinitrocresol with one and two mols. of dicyanodiamide have been isolated; of these, trinitroresorcinol (styphnic acid) seems to give a more nearly quantitative precipitation and better results than picric acid.

North-Western Cyanamide Co.,  
Odda, Norway.

#### NOTE ON THE DETERMINATION OF UREA IN FERTILISERS.

BY ERLING S. JOHNSON.

The method used in this laboratory for the determination of urea in fertilisers and fertiliser mixtures depends on the well-known fact that urea forms a characteristic, slightly soluble salt with oxalic acid. By using the necessary precautions the solubility of the oxalate can be made so small that a quantitative determination is possible. The following method was developed: 2–5 g. of the dried sample containing urea is extracted with 100 c.c. of anhydrous amyl alcohol, 25–50 c.c. of the filtrate is treated with an equal volume of ether, and the urea precipitated as oxalate by the addition of 25 c.c. of a freshly prepared 10% solution of anhydrous oxalic acid in amyl alcohol. The vessel is stirred and allowed to stand in cold water for half an hour and the precipitate collected on a Gooch crucible, washed first with a mixture of equal volumes of amyl alcohol and ether, and then with ether alone, the volume used in each case being about one and a half times the capacity of the crucible. The precipitate is dried in a vacuum desiccator and weighed. It contains 26.67% of nitrogen and 57.01% of urea according to the formula  $H_2C_4O_6 \cdot 2CO(NH_2)_2$ .

The appended table shows the necessary corrections as regards solubility:—

Urea found.	Percentage of actual amount present.
g.	
0.006	90
0.01	92.8
0.02	95.2
0.04	97.5
0.06	98
0.08	98.8

In some cases the urea is present as a salt or in complexes such as  $Ca(NO_3)_2 \cdot 4CO(NH_2)_2$ , in which

#### NOTE ON THE ESSENTIAL OIL FROM THE LEAVES OF *SKIMMIA LAUREOLA*.

BY JOHN LIONEL SIMONSEN.

*Skimmia laureola*, Hook. f., is an extremely aromatic evergreen shrub which grows throughout the temperate Himalayas from Murree to Mishmi and Khasia Hills. It is also a common undershrub in the Dun Hills. The oil obtained by the steam distillation of the leaves was found to consist mainly of *l*-linalyl acetate, whilst in addition a hydrocarbon, *l*-linalool, and a complex mixture of sesquiterpene alcohols and esters were isolated.

##### Experimental.

The oil used in these experiments was obtained by the distillation in steam of the shade-dried leaves, a steam pressure of 25 lb. being maintained during the distillation. The yield of oil averaged about 0.5% on the dry leaves.\* The oil, which was pale emerald green in colour, had the following constants:—Sp. gr. at 30°/30° 0.9041,  $n_D^{20}$  1.4648, acid value 3.63, saponification value 197.96, saponification value after acetylation 238.6.†

After washing with dilute alkali to remove the traces of free acid present, the oil was fractionated under diminished pressure (200 mm.), when the following fractions were obtained:—145°–160°, 18.5%; 160°–165°, 5.7%; 165°–175°, 38.6%; 175°–180°, 7.3%; 180°–190°, 9.4%; 190°–240°, 13.8%; loss and residue (by difference), 6.7%. The fractions distilling above 175° C. were all more or less deeply coloured, the colour varying from emerald-green to deep indigo-blue. The higher boiling fractions also showed a beautiful blue fluorescence, but careful tests indicated the absence of methyl anthranilate.

On further prolonged fractional distillation under diminished pressure (200 mm.) the following fractions were ultimately isolated:—(I) 130°–140°, 0.46%; (II) 140°–145°, 4.6%; (III) 145°–150°, 0.61%; (IV) 150°–155°, 8.4%; (V) 155°–160°, 2.3%; (VI) 160°–167°, 2.0%; (VII) 167°–173°, 50%; (VIII) 173°–177°, 0.61%; (IX) 177°–182°, 1.05%; (X) 182°–190°, 1.2%; (XI) above 190°, 18.2%.

Fractions I, II, and III appeared to consist essentially of a hydrocarbon. On redistillation a

\* The yield of oil and also the ester content varied considerably with the time of collection. Leaves collected in the Chakras Division during June and early July gave the above mentioned yield of oil, whilst those collected at the end of July and early August contained a much lower percentage of oil. On the other hand, a sample of leaves collected in the middle of August gave a yield of 1% of oil.

† Prior to acetylation the oil was mixed with turpentine as recommended by Boulez (Bull. Soc. Chim., 1907, 4, I, 117).



considerable fraction was obtained boiling at 130°–135° (200 mm.) and at 178°–182° (895 mm.). Analysis gave C=85.5, H=11.8%, whilst the following constants were observed:—Sp. gr. (30°/30°) 0.859,  $n_D^{20}$ =1.471,  $[a]_D^{20}$ =-4.11. The boiling point suggested that the hydrocarbon consisted largely of terpinene, but attempts to confirm this were unsuccessful. Treatment with sodium nitrite and acetic acid in petroleum solution resulted in the production of only the merest trace of a nitrosite, which separated in fine needles melting indefinitely at about 130°; too little was, however, obtained for recrystallisation and analysis. When oxidised with potassium permanganate in ice-cold solution in the usual manner no trace of erythritol or *aa*-dihydroxy- $\alpha$ -methyl- $\alpha$ -isopropyladipic acid was isolated. The nature of the hydrocarbon present in the oil must therefore for the present remain undecided.

Fraction IV consisted of nearly pure *l*-linalool, since on treatment with phenyl isocyanate in the usual manner an excellent yield of linalyl phenylurethane, m.p. 65° C., was obtained.

Fractions V and VI were found to be a mixture of *l*-linalool and *l*-linalyl acetate. After hydrolysis with alcoholic potassium hydroxide an oil was obtained which boiled constantly at 150°–152° (200 mm.) and showed all the properties of linalool. Acetic acid was isolated from the alkaline solution.

Fraction VII, which comprised the bulk of the oil, distilled mainly at 112° (20 mm.), and had the following constants:—Sp. gr. (30°/30°) 0.892,  $n_D^{20}$ =1.4537,  $[a]_D^{20}$ =-7.25°. (Found C=73.9, H=10.1; calc. C=73.5, H=10.2%). This fraction was pure *l*-linalyl acetate, as shown by hydrolysis, when *l*-linalool and acetic acid were obtained.

Fraction VIII gave on analysis C=74.4, H=10.0%, and was evidently nearly pure linalyl acetate. This was confirmed by hydrolysis when linalool and acetic acid were obtained.

Fraction IX, like fraction VIII, also consisted essentially of linalyl acetate. On hydrolysis linalool contaminated with a trace of a high-boiling alcohol and acetic acid were obtained.

Fraction X was not analysed. On hydrolysis it yielded acetic acid and a mixture of linalool and a high-boiling alcohol. The latter was not obtained in sufficient quantity for examination.

Fraction XI, boiling above 190° (200 mm.), was repeatedly distilled at 43 mm., but no constant boiling fraction could be isolated. Analyses of the various fractions, which were all deeply coloured, showed a carbon content varying from 76.1 to 81.0% and a hydrogen content varying from 10.3 to 11.2%. No more satisfactory results were obtained after hydrolysis of the various fractions, and further investigation of the somewhat unpleasant smelling oils was abandoned.

In conclusion the author wishes to thank Mr. Puran Singh for placing the results of the preliminary experiments at his disposal, and his assistant, Mr. T. P. Ghose, for making most of the analyses.

The Forest Research Institute,  
Dehra Dun, India.

#### NOTE ON THE PRODUCTION AND TESTING OF ZIRCONIA.

BY W. R. SCHOELLER, PH.D.

Several of the processes which have been proposed for extracting zirconia from Brazilian ore are based on the precipitation of zirconium as a basic sulphate (*cf.* Rossiter and Sanders; J., 1921, 70 T). The chief aim of these processes is the elimination of iron, generally without regard to the simultaneous

removal of any titanium present. Thus, Rossiter and Sanders' method, though shown to effect a quantitative precipitation of zirconia free from iron, admittedly fails to eliminate titania. This is not surprising, considering the readiness with which titanium sulphate is hydrolysed in a hot, feebly acid solution. It is remarkable, however, that the Brazilian ore experimented upon by Rossiter and Sanders was found to contain only "traces of titanium," and this statement may convey an erroneous impression that the ore from the Brazilian deposits is almost free from titania. Analyses of samples representing large shipments proved titanium to be a regular constituent of the Brazilian ore in quantities varying from 0.6 to 1.2%. The principal constituents of six samples are given in Table I; the analyses were carried out according to the method of Powell and Schoeller (Analyst, 1919, 44, 397; J., 1920, 67 A). Small amounts of lime and magnesia were also present.

TABLE I.

	A.	B.	C.	D.	E.	F.
ZrO <sub>2</sub>	79.58	77.47	76.13	68.82	56.56	53.50
SiO <sub>2</sub>	14.70	10.32	15.68	21.30	23.11	28.04
TiO <sub>2</sub>	0.63	0.98	1.29	1.12	0.65	1.04
Al <sub>2</sub> O <sub>3</sub>	1.73	1.53	1.45	2.62	3.61	6.06
Fe <sub>2</sub> O <sub>3</sub>	2.48	5.60	4.61	3.48	7.76	9.22
MnO	0.12	n.d.	0.39	0.19	0.25	n.d.
Ign. loss	n.d.	n.d.	n.d.	2.14	2.84	1.96
	99.34	95.95	99.60	99.57	99.28	96.82

n.d.=not determined.

Titanium and all other impurities found in the ore are, however, completely removed in the process based on the preparation of crystallised zirconium oxychloride. This process is seldom mentioned in English technical literature, probably because it is more expensive than a precipitation process; further, the oxychloride mother-liquor is fairly rich in zirconium, part of which is not recoverable in the pure state, because a solution heavily charged with impurities will not crystallise; but the operation is a simple one, and can be made to yield chemically pure zirconia.

It occurred to the author that the economics of a combined oxychloride-basic sulphate process might repay investigation, and the following suggestions for a process embodying Rossiter and Sanders' work are put forward tentatively. The finely ground ore is fused with sodium carbonate (*cf.* U.S. Pat. 1,261,943; J., 1918, 339 A), which the author considers superior to caustic soda because a better decomposition of the zircon in the ore is effected at the higher temperature required to melt the charge. The melt is leached 2 or 3 times with hot water, the volume of the first leach liquor being adjusted so as to deposit sodium carbonate crystals on cooling; the recovered alkali is available partly for neutralisation, and partly for opening up a fresh charge of ore. The insoluble residue is treated with the zirconium oxychloride wash-liquors and part of its mother-liquor, the solution evaporated to dryness, and the mass extracted with hot water. The filtered liquid is concentrated somewhat, mixed with hydrochloric acid, and left to crystallise; the crystals are filtered off by suction and washed with cold hydrochloric acid (1:1) till the washings are colourless. Instead of converting the oxychloride into zirconia by precipitation with ammonia and ignition of the hydroxide, the crystals may be heated in porcelain or silica vessels until the water and acid are expelled; the dry residue is powdered, boiled with dilute ammonia, filtered off and strongly ignited,

giving pure zirconia. The acid used in washing the oxychloride is mixed with a certain proportion of the oxychloride mother-liquor and used for dissolving the next batch of leached fusion residue; the balance of the mother-liquid—sufficient to prevent undue accumulation of impurities—is withdrawn from the circuit, neutralised with recovered sodium carbonate, and precipitated with sulphuric and sulphuric acids according to Rossiter and Sandera's process. The last operation yields the remainder of the zirconia practically free from iron and alumina, but contaminated with titania; this oxide is probably quite pure enough for certain industrial purposes (e.g., as a pigment). Further experimental work based on fractional solution or precipitation will probably indicate a method for eliminating the titania.

#### *Examination of commercial zirconia.*

To judge from the results of analyses of zirconia made by the author, the efforts of British manufacturers to produce a pure oxide have not so far been attended with success. The composition of the samples indicated that the ore had been opened up by fusion with nitre cake: the material was found to be strongly contaminated with arsenic pentoxide, no doubt derived from nitre cake obtained from impure sulphuric acid. The presence of arsenic is highly objectionable, especially where the zirconia is intended for use in X-ray work or as an opacifier in enamelling cooking utensils; it is liable to be overlooked unless a special test is made. For its detection and determination, 2 g. of zirconia is fused with 15–20 g. of sodium carbonate in a

platinum crucible, the melt is leached with hot water, the solution filtered, acidified with hydrochloric acid, and saturated with hydrogen sulphide. To expedite the precipitation of the arsenic sulphide, the acidified filtrate may be first digested on a water-bath with potassium iodide, which reduces the arsenic acid. The sulphide is filtered off, evaporated with sulphuric acid until fumes are evolved, the liquid made alkaline with bicarbonate and titrated with iodine solution.

Table 2 shows the impurities found in three samples of zirconia of British manufacture.

TABLE 2.

	(1).	(2).	(3).
SiO <sub>2</sub>	1.28	0.76	0.64
TiO <sub>2</sub>	1.45	0.98	1.70
Al <sub>2</sub> O <sub>3</sub>	0.16	0.30	0.13
Fe <sub>2</sub> O <sub>3</sub>	0.36	0.34	0.28
As <sub>2</sub> O <sub>3</sub>	1.03	1.37	1.30
SO <sub>3</sub>	0.16	1.85	1.94

In addition to the above constituents, the author has detected other impurities or added ingredients in specimens of zirconia obtained from abroad; viz., lime, soda, fluorine, chlorine, carbon dioxide, and boron trioxide. The analysis of zirconia must be made on the unignited sample, as the loss on ignition represents sulphur trioxide, carbon dioxide, fluorine, and chlorine, if present, as well as moisture or combined water.

## London Section.

Meeting held at Burlington House on May 2, 1921.

MR. JULIAN L. BAKER IN THE CHAIR.

### THERMAL LOSSES IN THE GAS PRODUCER PROCESS.

BY N. E. RAMBUSH.

The object of the present paper is to elucidate the principles by means of which it is possible to determine with a reasonable degree of accuracy the thermal efficiency of the gasification of a given fuel without large-scale tests, and generally to emphasise the relative importance of the various causes of thermal losses to engineers and chemists concerned with the working and design of gas-producer plants.

There are many types of gas-producers on the market, and within limits, it would be more or less right to say that producer-gas can be made in practically any apparatus. The object of this paper is not to discuss the advantages or disadvantages of any of the many producer types available, but it will be assumed that a gas-producer is employed which is suitable for dealing with the particular fuel and purpose under consideration.

Gas-producers and plants may be divided into two main groups:—

(1) *The hot gas-producer* in which the gas made is consumed in the same hot crude state in which it leaves the producer.

(2) *The cold gas-producer*, to which gas cooling and cleaning plant is attached to enable the gas to be supplied cold and of a degree of cleanliness that corresponds to the application in question. This group may again be divided into two sub-sections according to whether by-products are recovered or not.

The points having a bearing upon the thermal efficiency of the gas-producer process are:—(1) Moisture content in fuel, (2) nature of volatile matter in fuel, (3) grading of fuel, (4) soot formation, (5) ash content of fuel, (6) the radiation and convection heat losses from the producer proper to the atmosphere, (7) gas leakage losses, (8) losses in washing liquors, (9) moisture content in gas, (10) the temperature of and sensible heat contained in the hot gas leaving the producer.

Before dealing with these various points I should like to make clear the basis upon which I have determined the thermal efficiencies and losses. I have come to the conclusion that the best standard from the point of view of the gas-producer technologist is the ratio between the net heating value and volume at N.T.P. of the gas evolved per unit weight of theoretically dry fuel and the net heating value of the said unit weight of theoretically dry fuel. For plants in which the tar and oils are recovered as a valuable by-product and not considered a nuisance in the gas-making process, it might be argued that the heating value of the tar obtained (which can be utilised as fuel) should be added to that of the gas. Since the tar (especially on a by-product plant) may have a larger value to the industry than that corresponding to its heating value, I have refrained from doing so. On the other hand, it appears undesirable to complicate still further the matter of calculating the thermal efficiency of the gas-producer process by making allowances for the fuel required to raise the steam for the air blast, and create the power required for driving the auxiliary machinery, since this involves

factors entirely outside the proper process of gas making.

1. *Effect of moisture content in fuel.*—Most good-class bituminous fuels have such a low moisture content that the evaporation of this small amount of moisture has very little effect upon the thermal efficiency of the gas-producer process; but for such high-moisture fuel as wet coke breeze, and especially some peats or lignites, the moisture content will have a very great effect.

This may be demonstrated by the example of the gasification of a peat having a moisture content of 50% and a net heating value of 4000 Cals. per kg. (7200 B.Th.U. per lb.) of theoretically dry substance. For each kg. of dry substance of such peat introduced into the producer 1 kg. of water would have to be evaporated and heated to the gas outlet temperature of, say, 150° C.

Supposing now the peat instead of being introduced in a wet state were introduced in a moisture-free state into a by-product recovery producer, and the gas volume (at N.T.P.) and average specific heat of the gas evolved be approximately 2.5 cb. m. per kg. and 0.3 Cal. per cb. m., respectively, also that the gas outlet temperature be about 500° C., then the sensible heat in or the thermal capacity of the gas from 1 kg. of dry peat between 150° and 500° C. would be  $2.5 \times 0.3 \times (500 - 150) = 262$  Cals.

Now the heat required to raise 1 kg. of water to 100° C., evaporate it and superheat the steam to 150° C. will be 652 Cals., so that there is a deficit of  $652 - 262$  Cals. (=390 Cals.) per kg. of dry fuel, which must be supplied by burning a part of the fuel itself (roughly 10%) in the lower part of the producer, with a consequent reduction in the heating value of the gas and the thermal efficiency.

If the fuel bed is not deep enough to enable the sensible heat of the gas generated in the lower part to be utilised for the evaporation of the moisture, the thermal loss will of course be still higher, but the above remarks will indicate quite clearly why wet peat gasifications in the past have often met with so little success.

Returning now to the effect of the moisture content upon the thermal efficiency of the gas-producer process, it is obvious that before being able to estimate accurately the effect of evaporating the moisture it would be necessary to determine the difference in the gas outlet temperatures theoretically when using a dry and a moist fuel. Such determination could only be made by those practically experienced in this work, and even then may be liable to considerable error if the particular gas-producer design is not borne in mind.

As the object is to find what may reasonably be expected as to thermal efficiency, it is suggested that the heat required for bringing the moisture content to 100° C. and evaporating it be taken as having to be supplied from the heating value of the dry fuel only, while the heat required for superheating the steam thus raised is assumed to come from the sensible heat of the gas. Under this assumption the decrease in thermal efficiency obtainable in practice due to moisture will always be smaller than the thus estimated figures.

The curves in Fig. 1 show the reduction in the percentage of the thermal efficiency for fuels of varying heating value and different moisture content, estimated in this manner.

2. *Effect of volatile matter in fuel.*—Since the percentage of volatile constituents in fuels used for gas production varies from, say, 1% (on coke and anthracite) to above 80% (for woods), it is difficult to predict from the ordinary methods of ultimate gas analysis how much of this volatile matter will be obtained as gas or tar, unless of course the same material had been treated in a gas-producer before.

For the above reasons I decided about 12 months

ago to alter the method of fuel investigation in our laboratory at Stockton-on-Tees. The method I instituted is, generally speaking, to subject a small quantity of the fuel to a retorting test under temperature conditions approaching those obtained when treating the fuel in a by-product producer.

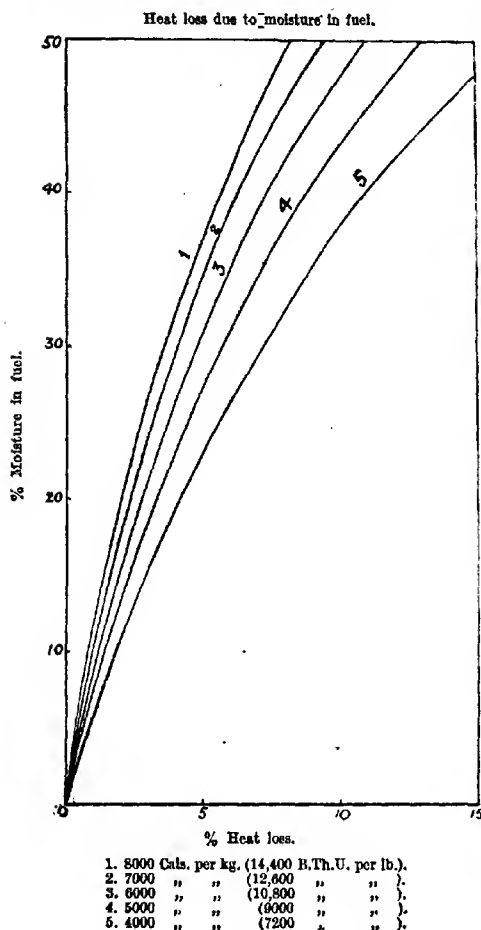


FIG. 1.

So far a reasonably close agreement has been found between the quality and quantity of the volatile matter obtained as tar in our special distillation test apparatus and that obtained in practice, excepting that the tar obtained in practice seems to have a higher average boiling point and is always obtained in a slightly smaller quantity. Probably some decomposition of the lighter oil constituents of the tar takes place in the producer. Our fuel testing method and its bearing upon the results to be obtained in practice is not yet completely established, but in another six months or so it is hoped to publish laboratory results, and also to show comparisons between these and the results obtained in practical operation with the same fuel.

As certain fuels in properly designed by-product producers may yield a very large quantity of tar, and as this tar per unit weight may have double the heating value of the fuel treated, a retorting test is, in my opinion, the best method by which to determine the "tar value" of a producer. Even if this should yield a slightly higher quantity of tar

than is obtainable in practice, the laboratory results will indicate a slightly lower thermal gasification efficiency than actual operation, and incidentally is far cheaper to carry out than a large-scale commercial test.

To arrive at the net heating value of a fuel from its ultimate analysis, all the oxygen is commonly assumed to be combined with the hydrogen. This is not so, since in practice substances like acetic acid, phenols, etc., are often obtained in the tar and liquors on a gas-producer plant, thus indicating the presence of carbon-oxygen links as well as hydroxyl groups in the fuel. For this reason, and since heat may be required inside the fuel itself to liberate its various particles, it is clear that a calorimeter determination, deducting the latent heat of the steam formed by the combustion of all the hydrogen of the fuel, be used as the measure for the net heating value instead of a net heating value arrived at from the ultimate fuel analysis.

3. *Effect of fuel grading.*—The grading of the fuel is a most important factor, not only from the point of view of the solid fuel particles that may be carried away by the gas (as dust), but also from the point of view of the output of the producer and the gas quality.

With fuels containing an excessive quantity of fines the dust loss may be serious unless preventive provision is made in the design. With the same grading of the fuel the loss is obviously a function of the gas velocity in the outlet pipe. Since the dust-carrying power of a gas alters with the sixth power of the velocity it is obvious that the best means to prevent thermal loss on this account is to have the crude gas as it leaves the producer as cold as practicable, or to provide large dust settling spaces (say a roomy space above the top level of the fuel bed).

To obtain the same gas analysis and output from fuels of various grading in a producer of the same diameter it is necessary to alter the fuel depth, or alternatively to provide that depth of fuel bed which is the most economical for the worst conditions of grading.

I have had communicated to me privately the following results of tests made in reference to this matter.

Fuel.	Grading.	Economical depth.
Coke .. ..	1 in.—1½ in. cubes	2 ft. 6 in.
	1½ in.—2 in. cubes	3 ft. 0 in.
	2 in.—3 in. cubes	5 ft. 10 in.
Coal .. ..	1 in. nuts	1 ft. 10 in.
Coal .. ..	Run of mine	4 ft. 10 in.—6 ft. 6 in.

Analyses of the fuel and of the gas are not given. It is, however, not surprising to find that the more uneven the grading and the larger the biggest pieces of fuel, the greater must be the fuel depth for efficient gasification.

It should be borne in mind that certain fuels (in particular some lignites) break up and crumble shortly after introduction into the producer, so that a larger dust loss will be obtained in practice than might be expected from the grading test carried out on the fuel in its natural state.

Other aspects to be considered, quite apart from the gas-producer process in itself, are the losses such as may be incurred (due to wind) by storing a slack fuel containing a large quantity of fines in the open or handling it in an unprotected fuel-conveying plant. Gas producer users will be well advised to have their fuel and storage plant protected against atmospheric effects, not only from the point of view of dust loss, but also on account of the moisture added to the fuel on rainy days.

4. *Soot formation.*—This varies with the type of fuel used, but it is caused by the tarry matters evolved from the coal being exposed to such a high

temperature inside the producer that they are decomposed into gas and carbon (soot). Soot formation is practically absent in some by-product producers where the only decomposition of tar which takes place is into gas and oils of a higher boiling point; but even with hot gas producers soot formation can be reduced considerably by feeding or distributing the fuel evenly over the top of the fuel bed, especially if the latter be not too shallow.

**Thermal loss by dust and soot formation.**—As is obvious this will vary considerably, but tests carried out several years ago on some large by-product producer plants working with rough slack as fuel indicate that this loss may be as high as 3% of the heating value of the fuel. With the latest designs of by-product producer plant it is reduced to a very much lower figure.

5. **Effect of ash.**—The three main factors in connection with the ash which have a bearing upon the gasification process are:—(a) the composition of the mineral matter in the fuel; (b) the sensible heat lost in the hot ashes; (c) the loss due to unburnt carbon in the ash.

(a) Except for the work by J. T. Dunn (J., 1918, 57) I am not aware that any considerable work has been done in reference to the fusing temperatures of ash from various English coals, although a large amount of work has been done both by the U.S. Bureau of Mines on American fuels and by the University of Zürich on Continental fuels. Until more extensive research has been carried out it will not be possible to point out in advance what quantities of steam would be the most advantageous to use or whether the producer should be water-jacketed or not, etc., in gasifying fuels of various qualities of ash. I shall therefore not go into the details of the extensive subject of ash fusibility, but only point out one or two other points of interest to the gas-producer technologist.

If the mineral matter in the fuel is not completely oxidised, extra oxygen is required from the air blast to complete the oxidation, with a consequent dilution of the resulting producer gas by nitrogen.

On the other hand, the ash may contain compounds which partly volatilise on heating up (say carbonates), also with dilution of the gas as a consequent result. Discrepancies in some fuel analyses are often due to similar causes.

(b) The sensible heat contained in the hot ashes leaving the producer varies with the ash content of the fuel, but is not of any consequence in a water-jacketed producer, because the heat is utilised practically completely to raise steam (by diffusion) and to preheat the air blast, but even with a "dry bottom" producer this heat loss is very small. Assuming the temperature of the ashes leaving the producer to be 100° C. and 400° C. for wet and dry bottom producers respectively, and assuming that 20% of the coal is removed as ash (sp. heat 0.3) the total sensible heat in the ash per kg. of fuel will be 6 Cals. and 24 Cals. and the loss of heating value of fuel (6700 Cals. per kg.) will be 0.09% and 0.36% respectively.

(c) The thermal loss due to incompletely burnt fuel varies, apart from the skill of the operator, with the heating value and the ash content of the fuel.

The figures in the following table give the approximate thermal loss due to this cause for several assumed conditions.

It is generally found in practice that the higher the ash content in the fuel, the higher will be the carbon content in the ashes; hence the ash loss is of the cumulative order in regard to the ash content.

It will probably be agreed that with bituminous coals containing up to 15% of ash it is possible to maintain the carbon content in the ash at or below 10%, which means that the thermal loss will not be more than 2.05% of the heating value of the fuel.

The carbon loss in % on the fuel used is calculated by the formula  $X=ac/(100-c)$ , where  $X$  is the carbon lost in percentage on fuel used,  $a$  the ash content of the fuel, and  $c$  the carbon content of the ash.

	Carbon content of ash, %	Lt. of carbon lost per 100 lb. of fuel gasified, i.e., % carbon loss.	Thermal loss on heating value, %
Fuel, 10% ash, 12,000 B.T.U./lb. or 6700 Cals./kg.	10	1.1	1.33
	15	1.8	2.17
	20	2.5	3.02
Fuel, 20% ash, 10,700 B.T.U./lb. 5950 Cals./kg. . .	12	2.7	3.46
	18	4.4	5.92
	25	6.7	9.05
Fuel 30% ash, 9500 B.T.U./lb. 5280 Cals./kg. . .	15	5.3	6.08
	23	8.1	12.85
	30	12.0	19.70

Fig. 2 has been prepared to cover the possible conditions met with in practice. Given a fuel of a certain ash content, and knowing the carbon content in the ashes, the thermal loss in terms of percentage on the net heating value of the fuel is at once obtainable.

6. **Radiation and convection loss.**—To obtain a more definite idea as to the order of the radiation and convection loss from the outside shell of a producer, I measured in 1918 the "skin" temperature of a 10-ft. by-product recovery gas-producer under full load conditions and found that the temperature 4 ft. from ground level was 69° C.; 6 ft., 85°; 8 ft., 101°; 10 ft., 109°; 12 ft., 92°; 14 ft., 79°; 16 ft., 76° C. The producer was 20 ft. high and the grate level was at 5 ft. 6 in. and fuel level 13 ft. from ground level. Thus the producer in question had an average skin temperature of about 80° C., while the total exposed surface was about 84 sq. m. Assuming for exposure to average atmospheric conditions a total radiation and convection loss of 760 Cals. per sq. m. per hour, we get a total loss of 63,000 Cals. per hour. The producer in question gasified about 850 kg. of coal per hour at 6700 Cals. net heating value per kg., hence the loss was equivalent to 1.1% of the heating value of the fuel. The producer in question had an exposed surface unnecessarily large in reference to its diameter, the exposed surface of many producers of the same or larger throughput, especially where no by-product recovery is aimed at, being only about 45–50 sq. m., with a corresponding decrease in the radiation etc. loss.

The radiation and convection loss from the outside casing of most gas producers to the atmosphere will be independent of the rate of gasification and represents a practically constant hourly loss proportionate to the total exposed surface.

Judging from the above practical observations as to the "skin" temperature and when using good class bituminous fuel, I consider that for the larger producer capacities (working at not less than  $\frac{1}{2}$  load) the radiation and convection loss should not exceed 1% of the heating value of the fuel gasified per unit of time.

7. **Losses due to leakage** may be subdivided into (a) periodical loss due to poking and cleaning, (b) continuous loss due to leaky joints.

(a) Assuming a 2-in. hole be open on the top of a producer having a gas outlet temperature of 500° C., then the corresponding hourly loss will be 3000 cb. ft. when the gas pressure is 2 in. and 10,000 cb. ft. when the pressure is 20 in. These gas volumes correspond to cold and clean gas. Many poking and cleaning holes are larger than 2 in. On many static producers a man will occupy on an average about one-tenth of his time poking the fuel bed of a producer, hence the thermal loss may easily

be 0.5% of the fuel gasified. Of course, on producers having mechanical stirring and ashing means this loss is practically eliminated. Cleaning of mains during operation should be avoided as far as possible, and mains should be arranged vertically or inclined so that they are as "self-cleaning" as possible.

(b) Leaky joints such as are caused by faulty manufacture or vibration (due to wind, machinery, etc.) exist even on the best plants. Even on such carefully laid pipe lines as for town gas supply it has been proved that leakages may be 1% of the gas supplied. Of course, the loss will depend on the surface of the plant exposed to the atmosphere, and

and thus escape to the atmosphere. The loss can be reduced, but its existence can be observed on any gas-washing plant by watching the behaviour of the washing liquid leaving a tower. If an inverted bottle filled with water is placed below the water level and close to the water outlet pipe in the tank, gas will soon rise in the bottle, replacing the liquid. From a test made by me on one of the early types of combined air saturator and gas cooler on a Lymn plant (by estimating the  $\text{CO}_2$  content in the air entering and leaving) the gas loss due to this cause was estimated at over 0.5%.

(b) All gases dissolve in water, and also some of the tar oil compounds are soluble, particularly the

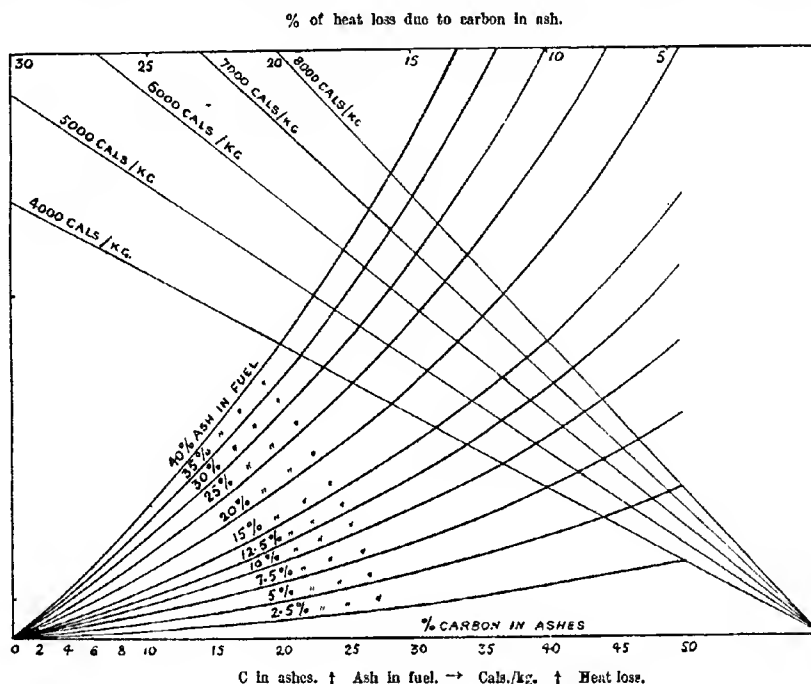


FIG. 2.

is obviously largest on cold gas producer plants. This type of loss becomes larger with cleaner gas, but smaller with lower pressure difference between the gas and the atmosphere.

It is obvious that the plant which works with the lowest possible pressure difference between the gas and the atmosphere will have the smallest leakage loss; thus if the whole plant were under auction this loss would disappear. For obvious reasons it is always advisable to have the gas under pressure at any points where poking or cleaning is required during operation. This pressure difference, however, should be maintained, automatically if possible, at the lowest limit.

Assuming average conditions I do not think that the leakage loss on a cold gas producer plant should be assessed at less than 1% of the heating value of the fuel gasified.

8. *Loss in gas cleaning and cooling plants.*—The losses on this account may be divided into two sub-headings, viz., (a) substances mechanically carried away by and (b) matters dissolved in the washing liquid.

(a) An appreciable quantity of gas is carried away mechanically with the washing liquors leaving the cooling, cleaning, absorbing, etc. towers

tar acids or phenols. The loss occurring in this respect, so far as the gaseous substances are concerned, is not very large and cannot be avoided; but by carefully controlling the waste liquors of the plant and the cooling water system, the phenols can be recovered. It is not impossible to obtain an effluent containing up to 0.5% of these phenols, which effluent can be economically treated. There are, however, many gas-producer plants in existence, where by exercising a little care and slightly modifying the working cycle of the plant the phenols can be recovered, instead of being run into the sewers.

Summarising the matter of the thermal losses due to gas carried away by the washing liquors, I do not think that even with a well-designed plant this should be considered less than 1% of the heating value of the fuel treated.

9. *Content of water vapour in hot crude gas.*—This originates from three sources: (a) That amount of the steam admitted with the air-blast which is not decomposed in the producer; (b) the amount due to "moisture content" of fuel; (c) the amount due to "water of decomposition" of fuel.

As to (a), the following figures, given partly in the two papers by Bone and Wheeler (Proc. Iron and Steel Inst., 1907 and 1908) and partly arrived



at in my own experience, will give a general guide as to the amount of steam decomposed in the gas-producing process when using bituminous fuels:—

Depth of fuel bed ..	3 ft. 6 in.				7 ft.				7 ft. 6 in.	12 ft.
Temp. of air blast, °C.	50	55	60	60	65	70	75	80	85	78
Lb. of steam per lb. of coal	0.21	0.33	0.45	0.45	0.56	0.80	1.1	1.55	2.5	1.45
% steam decomposed	95	100	78	87.4	80	61.4	52	40	36	62
Lb. of steam decomposed per lb. of fuel	0.2	0.33	0.34	0.39	0.45	0.49	0.57	0.62	0.75	0.60

It will be obvious that the deeper the fuel-bed the more complete is the steam dissociation. So far as the steam-carbon reaction is concerned it is obvious that the time factor plays a rôle.

I have further come to the conclusion that if by suitable modifications it is possible to increase the gasification capacity of a producer for the same fuel (such as by introducing mechanical stirring means), it is advisable at the same time to increase the fuel depth if a good quality gas is to be maintained.

Speaking broadly, and quite apart from the softening effect of the steam upon the clinker formation, the most economical blast temperature, from a thermal point of view, is that which corresponds to a practically complete dissociation of all the steam supplied with the air-blast. It has been calculated (see Richards, "Metallurgical Calculations") that the decomposition of about  $\frac{1}{2}$  lb. of steam per lb. of bituminous coal will counter-balance the exothermal reactions caused by the formation of the carbon dioxide usually present in gas obtained in ordinary hot gas producer work. It will be noticed that the calculated figure practically agrees with those quoted as results of ordinary practice.

From the point of view of the thermal efficiency of the gas-producer only (quite apart from the heat required to raise steam), it is obvious that the more undecomposed steam remaining in the crude gas, the larger will be the thermal loss due to the sensible heat in such undecomposed steam, which leaves at the same temperature as the gas. This rule holds good in spite of the fact that the gas temperature will be lower, the higher the percentage of undecomposed steam.

In connexion with the use of steam as the endothermal agent in the gas-producer process, reference may be made to a suggestion put forward many times in the past thirty years or more—namely, to introduce products of combustion instead of steam with the air-blast for the purpose of saving in the steam consumption. From the calculation below, showing the steam saving that can be effected at a certain reduction in the heating value of the gas, it will be clear why the experiments in this respect have been fruitless, and the gas in many cases useless for industrial work.

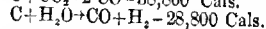
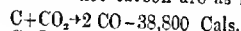
Assume that with the addition of steam only to the air-blast the following operating conditions are obtained:—Steam per kg. coal, 1.5 kg. (78° blast temp.); gas per kg. coal, 3.5 cb. m. (N.T.P.); heating value of gas, 1370 cal. per cb. m. (154 B.Th.U. per cb. ft.) net. Then steam per cb. m. of gas = 0.43 kg.

When adding products of combustion, assume that they have the composition: 18% CO<sub>2</sub>, 82% inert. Now assume that it is permissible to reduce the heating value of the gas by 20% (from 155 to 124 B.Th.U. per cb. ft.), due to the introduction into the gas of nitrogen from the products of com-

hustion. This means that if all the CO<sub>2</sub> is decomposed we can add per cb. m. cold gas:  $20 \times 100/82 = 24.4\%$  or 0.244 cb. m. of products of combustion.

The weight of carbon dioxide of this volume is:  $0.244 \times 18 \div 100 \times 1.964 = 0.086$  kg.

The relative endothermal effects of steam or carbon dioxide on hot carbon are as follows:—



—i.e., the endothermal effect of 1 kg. of carbon dioxide is equivalent to that from  $388 \times 18 \div (288 \times 44) = 0.55$  kg. of steam. Therefore the addition of 0.244 cb. m. of products of combustion to the air-blast will replace  $0.086 \times 0.55 = 0.0473$  kg. of steam.

In other words, the above rough calculation shows that by reducing the heating value by 20% the saving in steam is only 11% when using 1.5 lb. of steam per lb. of coal.

The calculation takes no account of the fact that the sensible heat loss incurred by cooling the hot gas is practically proportional to the volume of the hot gas, hence when adding products of combustion with a consequent larger gas production per unit of fuel the thermal gasification efficiency will certainly be less than with steam as the endothermal agent.

When the trouble involved in handling products of combustion is borne in mind, it is obvious that only disadvantages are to be looked for, unless perhaps the future enables us to use a gas richer in carbon dioxide.

As to the moisture content in the hot gas due to moisture in fuel and water of decomposition, both of these can be judged from the laboratory examination of the fuel, it being practically certain that none of the "moisture" in the fuel will be decomposed by the reaction with the carbon in an up-draught type of gas producer.

10. Effect of gas outlet temperatures and sensible heat loss.—If a good class bituminous coal is used the following gas composition and outlet temperatures may be reasonably expected:—

	Hot gas producer.	Cold gas producer.	
		Without recovery.	With ammonia recovery.
<i>Gas analysis:</i>			
CO <sub>2</sub> .. ..	5	9	10
CO .. ..	27	22	11
CH <sub>4</sub> .. ..	4	3	3
H <sub>2</sub> .. ..	12	20	26
N <sub>2</sub> .. ..	52	46	44
Moisture, g. per cb. m. of dry gas at N.T.P. ..	30	110	430
Equiv. saturation temp. of gas, ° C. ..	28	50	73
Temp. of hot gas ..	950° C.	700° C.	550° C.
Net latent heat of gas at N.T.P. ..			
B.Th.U. per cb. ft. ..	165.9	156.7	142.3
Cals. per cb. m. ..	1476.6	1394.5	1266.5
Thermal capacity per cb. m. gas N.T.P. in Cals.	329	264	290

In calculating the thermal capacity of the gas (total sensible heat) I have used the figures given

by Holborn and Henning for diatomic gases, steam, and carbon dioxide, and by Langen for methane. Fig. 3 has been drawn (completed early in 1918) upon which thermal capacity per cb. m. (N.T.P.) of each gas has been plotted against the temperature. In the above figures for sensible heat in gas any sensible heat contained in tar, dust, and soot has been eliminated, as the thermal loss due to these will be very small in comparison with the heat loss in the gas. The carbon and hydrogen losses from these three sources are of another order and have been dealt with under their separate headings.

Thermal capacity in Cals. per metre<sup>3</sup> (N.T.P.)  
between 0° and T° C. at constant atmospheric pressure.

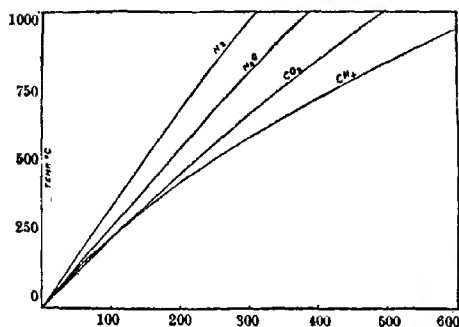


FIG. 3.

Sensible heat in hot gases.

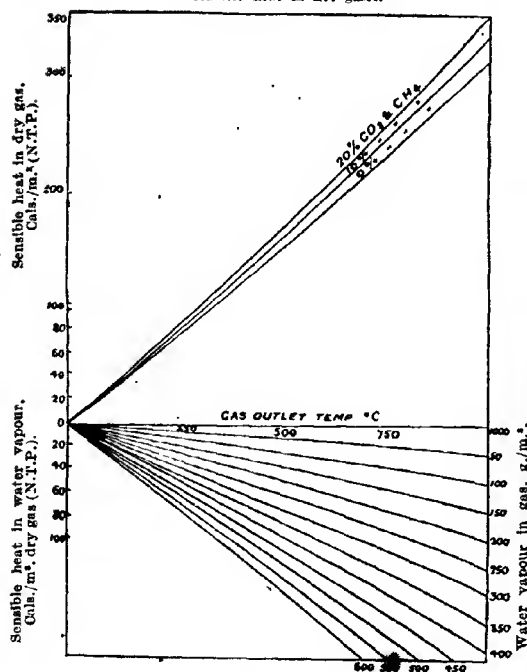


FIG. 4.

It will be seen from the summary table at the conclusion of this paper that in the three cases the thermal loss due to the sensible heat in the hot gases leaving the producer represent:—(1) 17%; (2a) 14%; (2b) 16% of the calorific value of the

fuel; therefore the sensible heat loss is the chief among all the losses of the cold gas producers. The only means available for utilising such heat is either to obtain a lower gas outlet temperature with a gas richer in heating value, or to utilise the heat for other purposes, e.g., to raise the steam required in the process by cooling the gas, either (1) directly by washing the air blast for the producers with the hot water from the gas cooling plant, or (2) indirectly by raising steam or heating water in boilers or vaporisers.

Loss due to sensible heat in gas.

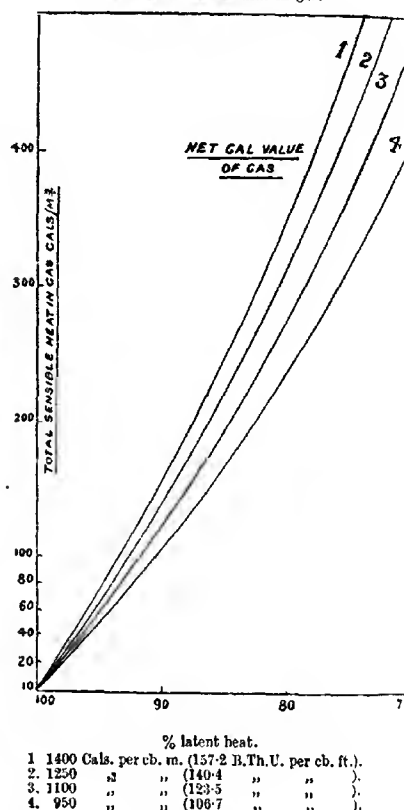


FIG. 5.

But even for the hot gas producers the loss of sensible heat in the gas may be of consequence if the furnaces are not placed close to the producers, and although it may be an advantage to have a high carbon monoxide content from a heating process point of view, it is often more economical from a thermal point of view to increase the hydrogen content somewhat at the expense of the carbon monoxide content, thus decreasing the outlet temperature and thereby radiation losses, while the difference in sensible heat for the two outlet temperatures will be present as the latent heat of the hydrogen in the gas.

It should therefore be emphasised that the gas composition is not always a measure of a thermally efficient hot gas producer plant unless the loss in gas temperature between gas producer and furnace is borne in mind.

All the above remarks are only generally applicable to an average English bituminous producer coal, but the gas composition, gas outlet temperatures, and moisture content in gas will vary

considerably with the various kinds of fuel gasified (from wood waste, wet peat, and town refuse to anthracite).

For this reason I have prepared Fig. 4 on which the sensible heat (thermal capacity) per cb. m. (N.T.P.) of gas of varying composition and of the accompanying moisture content can be directly determined for various gas outlet temperatures. Seeing that the methane content is generally less

than the carbon dioxide content, and its specific heat not very different from that of this gas and the rest of the components of producer gas (all diatomic gases), the diagram should prove of practical utility even if a small error is caused by assuming the specific heat of methane to be the same as that of carbon dioxide. I have also assumed that the relative specific heats of the various gases do not alter if the gases are mixed together.

	Types of plants and fuels.							
	Bituminous.				Anthracite.	Coke breeze.	Peat.	Wood.
	Hot gas.	Non-recovery.	Non-recovery.	Special recovery nts.	Non-recovery.	Non-recovery.	Recovery.	Non-recovery.
	1.	2.	3.	4.	5.	6.	7.	8.
<i>Laboratory examination.</i>								
Fuel analysis.								
Moisture .. ..	5	5	5	5	5	15	40	30
On dry sample—								
Ash .. ..	12	12	12	8	5	30	13	3
Volatile .. ..	30	30	30	31.5	5	6	50	71
T. carbon .. ..	72	72	72	80	87	64	50	47
F. carbon .. ..	55	55	55	55.5	—	—	34	24
Hydrogen .. ..	4.5	4.5	4.5	5.0	—	—	5.5	5.8
Net B.Th.U. per lb.	11,750	11,750	11,750	12,250	14,000	0,800	8,250	8,500
Fuel grading—								
Above $\frac{1}{2}$ in. ..	60	60	60	94	100	15	Lumps.	Blocks.
$\frac{1}{2}$ in. to $\frac{1}{4}$ in. ..	20	20	20	4	—	36	—	—
Below $\frac{1}{4}$ in. ..	20	20	20	2	—	50	—	—
% tar yield in small retort-	—	7	7	8.8	—	—	—	8.5
ing test .. ..	—	1.3	1.3	1.25	—	—	1.8	1.75
Ratio of cal. val. tar : fuel	—	—	—	—	—	—	—	—

#### Expected working results.

Cold gas analysis—								
CO <sub>2</sub> .. ..	5	9	16	11	7.5	7	30	11
CO .. ..	27	22	11	17.5	24.0	24.5	9	20
CH <sub>4</sub> .. ..	4	3	3	3.3	1.2	1.1	3.3	3.3
H <sub>2</sub> .. ..	12	18	26	21.5	16.5	11.0	21	16
N <sub>2</sub> .. ..	52	43	44	46.7	50.8	56.4	43.7	49.7
Net B.Th.U. per cb. ft.	185.9	156.7	142.3	154.4	141.8	126.7	124	146.9
Cals. per cb. m.	1470.5	1394.6	1266.5	1374.4	1262	1127.6	1103.6	1307.4
Outlet temp. of hot crude gas, ° C. ..	950	700	550	400	700	700	150	250
Moisture in hot crude gas, g. per cb. m. (N.T.P.) ..	30	110	430	150	40	90	600	450
% carbon in ash .. ..	10	10	10	10	20	20	12	10
Tar yield% .. ..	—	4	5	8	—	—	7.7	7.5

#### Assessment and calculated results of thermal loss in % of net heating value of fuel.

Moisture loss .. ..	0.5	0.5	0.6	0.5	0.3	2.2	9.5	6.0
Tar loss .. ..	Nil	5.2	6.5	10.0	Nil	Nil	14.0	13.0
Dust and soot losses ..	3.5	3.0	3.0	0.5	0.5	2.0	1.5	2.0
Ash loss .. ..	1.6	1.6	1.6	1.0	1.2	10.5	4.5	0.5
Radiation loss .. ..	1.0	1.0	1.0	1.0	1.0	1.2	1.5	1.5
Leakage losses .. ..	0.5	1.0	1.6	1.0	1.0	1.5	1.5	1.5
Total .. ..	7.1	12.3	14.1	14.0	5.0	17.4	32.5	24.5
Remaining in hot gas as total sensible and latent heat .. ..	92.9	87.7	85.9	86.0	95.0	82.6	67.5	75.5
Sensible heat loss as calculated below .. ..	Nil	14.0	16.2	9.4	15.2	16.3	8.2	8.3

#### Sensible heat loss in cals. per cb. m. of gas (N.T.P.).

% CO and CH <sub>4</sub> .. ..	49	12	19	14.3	8.7	8.1	23.3	14.3
% diatomic gases .. ..	91	88	81	85.7	81.3	91.9	76.7	85.7
Heat in dry gas .. ..	314	223	180	123	224	224	50	79
Heat in steam .. ..	15	36	110	23	14	30	42	51
Total sensible loss, cals. per cb. m. ..	329	264	290	156	238	254	92	130
Latent heat of gas .. ..	1476.5	1394.6	1266.5	1374.4	1262	1127.6	1103.6	1307.4
Total heat of hot gas, cals. cb. m. ..	1806.5	1658.6	1556.5	1530.4	1500	1381.6	1195.6	1437.4
Ratio—Latent heat .. ..	81.7%	84%	81.2%	89.2%	84%	81.6%	92.4%	89.0%
Thermal efficiency .. ..	92.9	73.7	69.7	76.6	79.8	67.4	62.3	67.2

\* This calculation is made only for comparison, and the loss only exists in practice in a degree corresponding to the difference in the gas temperature at furnace and leaving producer.

It may have been noted that all the losses in the gasification process, except the sensible heat loss, can be expressed in terms of the net heating value of the fuel. As, however, it is easier to express the sensible heat loss in terms of that contained per unit volume (at N.T.P.) of cold gas, it will be necessary when determining the expected thermal efficiency first to assess all the other losses, thus giving as a deficit that heat which must exist as sensible heat in the hot gas and as latent heat in the cold gas. When the ratio between the net available latent heat in the cold gas and the total sensible and latent heat in the hot gas is calculated, the sensible heat loss in terms of the calorific value of the fuel, and therefore the net gasification efficiency, can be obtained (see Fig. 5).

*General use of above statement.*—Following are some examples of how the above statements may be applied for calculating the thermal gasification efficiency of various types of fuel and plant. The figures and assumptions are given in the serial order in which they are best determined.

As regards cases 3, 4, and 5 the actual thermal efficiencies obtained in practice were:—(3) 70·2; (4) 74·5; (5) 76·5. The figure in case 3 represents a yearly average of data on coals used and gas metered, while case 4 represents a 288 hours' continuous test and case 5 a one-month test.

The discrepancies between theory and practice of the three cases are of so small an order that the method of calculation put forward is in my opinion sufficiently reliable for general adoption—perhaps in certain cases with an additional factor of safety.

In conclusion I wish to express my thanks to the Directors of The Power-Gas Corporation, Stockton-on-Tees, for their kindness in permitting me to publish results obtained by my firm, and also to Mr. A. T. Grisenthwaite, B.Sc., for help given in working out the curves and table appended hereto.

#### DISCUSSION.

Captain C. J. GOODWIN asked as to the possibility of utilising a small quantity of oxygen for enriching the air supply to producers. It would increase the temperature considerably and tend to fuse the ash, but he thought there was the possibility of obtaining improved results.

Dr. M. W. TRAVERS said that he had been working at the heat balance of the steam-blown gas-producer (*cf.* J. Soc. Glass Tech. 1921). From experience in connexion with the glass industry with modern gas-producers, from which the gas issued at a temperature of not over 700° C., it did not appear that soot was actually formed in the producer. The gas as it issued from the producer was loaded with tar fog, but the actual formation of soot appear to be, at least in some measure, due to the reversal of the CO—C—CO reaction in contact with the dust. In speaking of the velocity of the gas issuing from the producer he presumed that the author referred to the velocity of the gas in the top layer of the fuel bed. Some interesting work on the nature of the fuel bed, the resistance to the flow of the gas, and the nature and grading of the fuel had been carried out by the U.S. Bureau of Mines (Tech. Paper 137, p. 57). He had been unable to find any information in technical literature which would enable him to connect the chemical and thermal properties of tar, which was certainly endothermic. In one of the latest publications on the subject, the Report of the Research Committee of the Institution of Gas Engineers, it was assumed that the heat of formation of the tar could be calculated from the composition of the tar, on the assumption that the heat of formation was equivalent to that of naphthalene. Mr. Rambush had determined the heat of combustion of the tar, and a few analyses of his material would provide the necessary information.

Mr. P. PARRISH said that the author gave the thermal efficiency of a coke breeze gas-producer plant as 66·3% where cold gas had to be utilised; did he regard it as more advantageous to adopt the gasification of coke breeze in conjunction with a Bonecourt boiler for the production of steam in lieu of ordinary steam production in a Lancashire boiler using a forced draught furnace for coke breeze? In the latter case an efficiency of 58—60% was certainly possible. The cost of the plant, i.e., the producer for gasifying coke breeze and the provision of a Bonecourt boiler for an installation capable of dealing with 750 gallons per hour, would be something of the order of £6000, whereas a Lancashire boiler with a chimney shaft to deal with 750 gallons per hour would cost £3000. The theoretical efficiency which the author would lead one to expect was 66·3%, and that higher efficiency entailed an extra expenditure of £8000 for 750 gallons per hour. Did he consider that to be a commercial proposition which would justify embarking on the gasification of coke breeze in a producer in conjunction with a Bonecourt boiler, in comparison with the forced draught type of Lancashire boiler, which was in operation to such a large extent to-day? Again, he doubted whether it was possible economically to gasify coke breeze by itself. It had been tried at the Birmingham gas works for a long period, but it had been found necessary to mix 50% of producer coal to get satisfactory results. Moreover, in view of the flame temperature of the cold producer gas, it was found that some form of regeneration was necessary to get the best effect.

Mr. ARTHUR H. LYMN said that he had carried out a large number of large-scale tests during the last twenty years, and had seldom known them to come nearer the actual regular figures of later works practice than did the figures which the author had given. Consequently the author's anticipated results seemed so near the actual results of works practice that they not merely anticipated the figures of large-scale tests, but practically eliminated the necessity for them. The efficiency of the producer should include the steam that went into it, although in those cases where other apparatus was included with the producer, such as washing plant, it was always possible to recover that steam from the waste heat of the gases. Nevertheless he would like to see the figures in the paper also shown on the basis of including the steam. In dealing with the heat contained in the gas from hot gas-producers as compared with the recovery gas, the author had taken certain temperatures of the outgoing gases from the producer. His own opinion, from a wide experience, was that 900° C. was scarcely a fair figure for hot gas-producers; it should not be more than 700°, and where the author spoke of 17% in comparison with 14% losses for different types of plant, that would probably change this to 12 or 13%, but it would be well if they could have the calculations for an outgoing temperature of 700° also, because a well-conducted hot gas-producer should not have a higher temperature than that. The author had drawn attention to the determination of the fusibility of ashes. He (the speaker) suggested that some institution in this country should undertake a systematic investigation of the fusibility of fuel ashes, as had been done by Constam for German fuels some years ago.

Dr. R. LESSING agreed that the fusibility of ashes was one of the most important points in running gas-producers. The fusibility of the ash in a coal sample need not be exactly the same as that obtained when working on a large scale, because in the latter case they were not dealing with a finely powdered and homogeneous sample. In some samples of ashes of mixed fuels consisting of cannel and coals there was a large lump of refractory cannel ash enveloped in fused coal ash which had

held up the whole fuel charge and led to trouble. In that case a properly prepared sample would have shown a very high degree of refractoriness, but not fusibility. He had adopted a plan of carrying out ash analyses on an extensive scale by which it was possible to predict the clinker formation in a particular fuel charge with a high degree of accuracy.

Professor J. W. Coss wrote that any reliable process which enabled the individual losses incurred in producer-gas practice to be gauged with an approximation to truth from a laboratory examination of the fuel would serve a useful purpose. The loss due to sensible heat in gases leaving the producer was due to the carriage of heat upwards by the products of gasification of the fixed carbon in the bottom of the producer, and its incomplete extraction in the distillation process at the top; if, as now seemed likely, it became possible to replace air in many cases by oxygen in gasification owing to the cheapening of oxygen which was being brought about, the absence of nitrogen in large volume would tend to lessen that loss. Other accompanying economies would also increase efficiencies so far that some items of loss inevitably remaining would assume a greater importance relative to the total loss than had been the case in the past. One of these was the loss by radiation and convection, which had usually found a place in an omnibus item, termed "radiation, convection, etc.," and was made responsible for leakage, unknown losses, and the avoidable and unavoidable errors of experiment. He was interested therefore in the author's observations and calculations under this head. His figure of 1% for a large producer under at least three-quarters load confirmed the one which Mr. Hodsman and himself had felt justified (from the substantial agreement of three methods of calculation) in using when estimating the probable losses of an oxygen gasification process.

Dr. J. H. PATERSON wrote that from a theoretical point of view the losses which took place during the artificial cooling of the gas produced had little interest. On the other hand, data showing the relationship between gas outlet temperature and rate of combustion and thickness of fuel bed would add very considerably to our somewhat scanty knowledge of these subjects. Of great value also would be curves showing the effect of fuel bed thickness and rate of working on the unaltered water content of the gas, and also on the hydrogen content.

Mr. RAMEUSH, in reply, said that it was possible to design a gas-producer to use oxygen or air enriched with oxygen; probably it might mean a producer in which the ash was tapped out as molten slag, because (unless endothermal agents were added to a large extent) the heat would be far more intense in the lower zones. Investigations on the relative effect of steam and oxygen in the gasification process were still in hand. He had not been able to determine the heat required for liberating the tarry matter from the fuel, but considered it to be of a small order, since the fall in temperature of the gases between the lower and upper part of the producer would account for this splitting up; in other words, if there were no volatile matters to be driven off, the gas outlet temperatures would be still higher than those used in the tables in his paper. The amount of dust removed depended upon the velocity in the gas outlet pipe; if the velocity in the gas outlet pipe became lower due to the removal of a smaller volume of gas per unit of time, it would become lower in the upper parts of the fuel bed as well as in the gas space above. With regard to the efficiency calculated for the gasification of coke breeze, he had chosen as typical a coke breeze containing 30% of ash although he had gasified successfully coke ballast containing up to 45% ash. By careful working the ash loss might be made less than the 10% assumed as an illustration, but whether

such a coke breeze could be burnt more efficiently on a boiler grate he could not say. The coke breeze gas producer plant at the Birmingham Gas Works was designed for using coal, but as there was a superproduction of coke breeze to be burnt somewhere, it was burnt with the coal in the gas producers. Dr. Lessing had mentioned the clinker formation when using mixed fuels. His experience was that the use of mixed fuels often caused such difficulties and his advice was not to mix different fuels if the best efficiency were aimed at. In his opinion there was still more work to be done on the relative fusing temperatures of ashes from various coals, such as had been commenced by Dr. Dunn. The temperature of the outgoing gases was very important and depended upon many factors especially upon the depth of fuel bed and the quantity of steam that was being added to the air blast. He had seen gas producers with temperatures as high as 900° and others of 700° C.

In reply to Dr. Paterson's remarks, the losses that took place by artificial cooling were of great importance as they emphasized that a thermally economical cold gas producer must be so designed as to give a crude gas leaving the producer, which retained as sensible heat as little as possible of the heat of the solid fuel. The further data required in reference to depth of fuel bed in reference to all factors to be considered in the working of a gas producer plant were very difficult to put down in definite figures, unless given in reference to a given fuel of constant quality and grading.

Regarding Prof. Cobb's remarks there was a limit to the completeness of the extraction of heat from the hot gases leaving the producer and that limit depended either upon what one could afford to spend in the construction of the gas producer, or upon the possibility of gasifying the particular coal at very low temperatures. The present state of the industry did not enable one to lay down any definite rule in this respect.

## Glasgow Section.

Meeting held on February 22, 1921.

MR. J. H. YOUNG IN THE CHAIR.

### LEAD CORROSION WITH RELATION TO SULPHURIC ACID MANUFACTURE.

BY WM. G. McKELLAR.

In the manufacture of sulphuric acid by the chamber process, frequent troubles are encountered owing to the failure of the lead sheets used in constructing the chambers, and an attempt has been made to ascertain the most suitable quality of lead for the purpose. Two types of desilverised lead ore in general use for building chambers, namely, Pattinsonian and Parkes; each brand is advocated by the various refiners, and every maker guarantees that his lead is at least of 99.9% purity.

In the past Parkes's lead has often been regarded with suspicion, and generally for chamber building it has been blamed, I believe, by most chamber constructors, who prefer to use Pattinsonian lead; but in spite of preferences for one brand of lead or another, the results obtained, measured by years of service, out of any particular chamber or set of chambers are very unsatisfactory, and the reasons for failure most obscure.

It should be noted that while the problems relating to lead in chambers seem to be ever-recurring, trouble seldom occurs with lead used

in Glover towers, cisterns, gas flues, and pumping pipes. Lead used in these situations usually appears to be immune from the troubles which beset chamber lead.

When the conditions of chamber working are surveyed, the following points are seen to deserve attention.

**Method of supporting the leaden sheet.**—Some builders prefer to hang the sheets from the top rail of the chamber frame and have no cross rails in the structure; the leaden sheets are secured to the vertical posts simply by clinching or folding over the end of the strap round the section of the post. Now while this form of construction has the merit that it provides very efficiently for expansion and contraction due to changes of temperature, it creates a difficulty which does not seem to have been sufficiently appreciated, arising from the fact that the sheet at the point of suspension is in a state of stress due to the weight so carried, which stress is a maximum at the point of suspension, reaching zero at the bottom of the sheet; a sheet so hung (when the chamber is at work) has its interior surface attacked by a corrosive atmosphere, accompanied by a relatively high temperature,  $100^{\circ}$ – $180^{\circ}$  F. ( $38^{\circ}$ – $82^{\circ}$  C.), and these three sets of conditions, namely, stress and corrosion with high temperature, acting at the same time, appear capable of producing disastrous results in a very short time. Fig. 1 shows a strip cut from a sheet in a chamber, the entire lead work of



Top. Middle. Bottom.  
Lead sheet, showing aggravated corrosion at top of sheet.

FIG. 1.

which failed or gave way after a much shorter time than it should have done; the sheet is badly pitted, the pits being deepest and more marked at the top and disappearing towards the bottom. Analysis in such a case is useless, and even micrographical examination failed to disclose any difference in structure likely to cause this failure. The immunity from attack by sulphuric acid enjoyed by lead is probably due to the formation of an insoluble lead sulphate, and it can easily be conceived that if a protecting skin on lead once formed is subject to a continuous stretching action the skin will never be allowed to adhere, but will become detached as quickly as formed; further, the continuous stretching action exposes fresh lead surfaces to the action of the gases, and these surfaces are probably very susceptible to the action of the acid gases. There is therefore some reason in favour of modifying our views regarding this form of chamber construction. The alternative system of cross rails raises difficulties due to timber being an excellent non-conductor and not allowing sufficient heat radiation from the lead skin, and notwithstanding all the devices which have been proposed for helping to get the heat away, much yet remains to be done to perfect the cross-rail system. Further, the cross-rails prevent the free circulation of air round the chamber sheets, which is most desirable, and there is a lack of flexibility in the provisions for expansion. In any system in

which timber is in contact with lead work, the life of a leaden chamber is greatly lessened at these points of contact.

**Chamber ventilation.**—In any chamber plant working under normal conditions, there is always a large amount of energy in the form of heat due to the reaction between the sulphur dioxide and oxygen, which must be dissipated either by natural ventilation or by artificial cooling by water sprays on the outside of the lead work. Water cooling can be and is carried out satisfactorily, although this method involves an extra charge; but where there is no such arrangement, it is necessary to depend on the access of a free circulation of air round about the lead work. In the past this problem has not had adequate attention. In too many cases the passages are unduly restricted, due no doubt to the desire on the part of the builder to get as large an internal capacity as possible; in the west of Scotland the very wet climate is also a contributory cause. The passages are roofed in, thus imprisoning much heat that could easily be spared and tending to shorten the life of an acid chamber. In the years when lead cost £12 or less per ton, I believe it would have paid manufacturers to floor the chamber passages with lead, allowing free ventilation for the chamber sides and ends by dispensing with passage roofs.

**Efficiency of nitre recovery.**—With no system of nitre recovery, the quantity of sodium nitrate used would amount to 11% on the sulphur charged, but by the introduction of the Gay-Lussac tower this figure was reduced, till to-day over the United Kingdom it might average  $3\frac{1}{2}\%$ ; on certain plants figures as low as 1% are claimed, and with close supervision and adequate gas scrubbing plant there is no doubt this low figure can be obtained over a continuous period of months. A low nitre consumption or loss leads to an increased life in a chamber plant, and the reason is not difficult to find. If sodium nitrate is "potted" or ammonia oxidised, nitric acid is formed, and this enters the chambers and attacks the lead work, whereas if the nitrosulphonic acid is decomposed by mixing with weak acid, and the mixture run down the Glover tower, either  $N_2O_4$  or  $N_2O_3$  is formed, both of which are excellent carriers of oxygen without the vicious properties of nitric acid.

**Supply of water vapour.**—The regular and well-distributed supply of water vapour, either in the shape of water sprays or steam, is essential. If the supply is insufficient, the strength of the acid mist in a chamber may rise dangerously, and a set of conditions exist which cause serious destruction of the lead work. On the other hand, if the supply is in excess, equally serious damage may be done to the lead work, accompanied by a greatly increased loss of nitrous compounds.

For all the four factors dealt with above as having an influence on the life of a sulphuric acid chamber, the chamber management alone is responsible; factors which merit the attention of the lead maker will now be considered.

**Speed of sheet lead rolling.**—The work of the Corrosion Committee of the Institute of Metals has shown that the cracking of brass condenser tubes is due to the state of internal stress produced in drawing them out; in fact any form of stamped or cold-drawn brass work is unreliable because of the abuse it has received in the process of manufacture, and in the light of a failure of lead, to be described, it is evident that lead also can receive such abuse on rolling that it never recovers. It may be said that lead anneals at ordinary temperatures; but while this may be generally taken as correct, it would seem that there are cases in which the lead has been so severely treated that it did not recover even after the lapse of years. The particular failure to which reference is made happened both at our St. Rollox factory and in that of one of



our neighbours. A large number of leaden boxes were made for the purpose of sending sulphuric acid abroad; the lead was  $\frac{1}{4}$  inch thick and weighed 5 lb. per sq. ft. Each box carried 198 lb. of acid, and there was 7% clearance space to allow for any gas that might be given off after filling. The boxes were protected by an outside casing of wood, and all the joints were autogenously welded by means of the air and hydrogen flame. A small filling hole was cut out of the roofs of the boxes, and after filling the boxes were finally sealed by welding as before, a period of two days elapsing between the filling and final sealing, so as to allow of any gas generated to escape. The boxes lay in the warehouse for seven days preparatory to shipping, and about 3% developed leaks in a most alarming fashion. On examination of the defective cases it was found that numerous cracks had been formed in the lead, through which the acid flowed. The lead was exceedingly brittle, and a moderate blow with the fist on a defective side of a box caused the lead to give way with an irregular fracture, and left a hole which resembled the hole left in a glass pane when broken with a stone. There was no swelling of the lead work, so that it was evident that the lead had not been stressed by internal pressure. The lead was brittle and broke off short when bent in any direction. Exhaustive analyses showed that the lead was 99.9%.

It was evident that some means other than analysis was required to assist in clearing up lead problems, and accordingly I have endeavoured to inaugurate micrographic work for this purpose.

Naturally a lead maker wants to run his mill at the highest possible speed so as to get the highest output per unit of power and other charges involved, but care should be taken that the sheet of metal so produced has not received serious injury in the rolling process. Where cost is immaterial, certain architects insist on cast lead for roof work, and it is alleged that such cast lead is very superior to any form of rolled lead.

The presence of lead oxide inclusions in the mass of the metal is another source of failure, and these account, I believe, for the failures which take place in new leaden sheets in a chamber when first put to work. The failure takes the form of minute circular perforations which soon develop into large holes, and sheets having this defect never have a satisfactory life.

**Attempt to solve the problem of lead corrosion.**—Some fifteen years ago it was stated that lead containing about 0.1% of copper, either resulting from the presence of copper in the ores or introduced into the molten lead, resisted the action of the gases in a sulphuric acid chamber excellently. I regarded this very much as a quack remedy, and microscopical examination confirmed this, but in order to test the question along other lines four sample strips were prepared as follows:—

	No. 1. Zinc- treated lead with copper.	No. 2. Pure Pattinsonian.	No. 3. Zinc- treated lead.	No. 4. Pattinsonian with copper.
Pb (diff.)	99.9029	99.9307	99.9719	99.9142
Cu ..	0.0790	0.0006	0.0017	0.0780
Bi ..	0.0120	0.0004	0.0170	0.0006
Sb ..	0.0005	0.0017	0.0008	0.0012
As ..	0.0009	0.0009	0.0017	0.0008
Sn ..	0.0022	0.0023	0.0030	0.0025
Fe ..	0.0003	0.0017	0.0003	0.0007
Mn ..	NH	NH	NH	NH
Ni ..	NH	Tr. trace	NH	0.0004
Zn ..	0.0020	0.0020	0.0034	0.0015
Ag ..	0.0002	0.0003	0.0002	0.0001

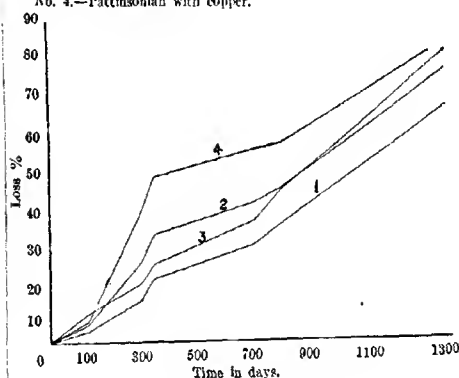
The strips measured 10 $\frac{1}{2}$ " $\times$ 3", and were hung facing the travel of the gases by leaden hooks

in the centre of a second chamber in a set for varying periods of time, and the losses in weight noted. The experiment lasted in all 1270 working days; at the conclusion the pieces had the following composition:—

	1.	2.	3.	4.
Pb (diff.)	99.8946	99.9796	99.9647	99.9021
Cu ..	0.0550	0.0010	0.0004	0.0660
Bi ..	0.0400	0.0110	0.0260	0.0255
Sb ..	0.0030	0.0018	0.0024	0.0019
As ..	0.0009	0.0004	0.0008	Trace.
Sn ..	0.0020	0.0031	0.0036	0.0025
Fe ..	0.0003	0.0012	0.0003	0.0006
Mn ..	NH	NH	NH	NH
Ni ..	NH	NH	NH	Tr. trace.
Zn ..	0.0020	0.0017	0.0016	0.0013
Ag ..	0.0002	0.0002	0.0002	0.0001

Unfortunately the analysis failed to throw any light on the problem, and the appearance of the pieces did not produce any evidence of value in determining the factors which regulated corrosion. The losses which occurred are shown in the following table and graph. The curves show to some extent the value of the clean, polished surface of the new

No. 1.—Parkes with copper.  
No. 2.—Pattinsonian.  
No. 3.—Parkes alone.  
No. 4.—Pattinsonian with copper.



Time, days. % Loss.

Time, days.	1.	2.	3.	4.
121	2.92	4.82	7.80	5.63
288	11.30	21.7	16.2	35.4
328	17.34	29.34	21.44	45.24
649	26.23	37.99	32.87	53.10
734	31.63	41.65	41.34	54.61
1270	64.69	74.72	79.86	82.21

lead in resisting attack, and also illustrate the protective value of the lead sulphate formed on the lead as indicated by the increased rise in the curve after cleansing at short intervals.

These curves also raise the query "if the addition of copper was of value in the case of lead No. 1, why was it not equally valuable in the case of No. 4?"

Some time after the test had been running it was evident that it would have been of great advantage if there had been four parallel samples under test, which were only weighed at the conclusion of the experiment, and the disturbing effect of washing eliminated, but the test had reached such a stage that this could not be done for want of sufficient sample material to work on.

Micrographs were made of all four samples. The samples were cut with a sharp joiner's chisel, washed with methylated spirit to remove grease and dirt, immersed in cold 10% nitric acid, and the lead nitrate removed at frequent intervals by means of a camel-hair swab brush. The etching occupied about 3–4 days.



Parkes lead (copper-treated).  
× 12.

FIG. 2.

It was found later on that the samples cleaned by spirit were often twisted and uneven, and as it was not desirable to flatten them out with a blow, they were planed true with a Stanley plane and then etched. A sample of copper-treated lead shows no outward indication of the presence of copper, but if it is etched the copper speedily appears as large segregates, which are apparently capable of causing a good deal of harm. Fig. 2 shows a sample of Parkes lead (copper-treated, ×12) etched by 1/1 nitric acid vapour, and reveals a deep pit with a large copper segregate, the deep black stain being due to a very dense segregate and the lighter shaded marks to less dense segregates.



Pattinsonian lead, copper-treated.  
× 80.

FIG. 3.

Fig. 3 shows a sample of Pattinsonian copper-treated lead (×80) etched with 10% nitric acid; the black network showing all through is due to streaks and prills of copper. When 10% nitric acid is used for etching it is very difficult to keep the copper from disappearing, but by etching with the vapour from 50% nitric acid the copper is not nearly so severely attacked; unfortunately lead recrystallises at about 90° C., so that the vapour

method of etching is of no use in examining the general lead structure.

From Figs. 4–7, showing the four sample pieces etched with 10% nitric acid, it is evident that the addition of copper to lead causes a very marked reduction in the size of the crystals. This may, however, be due to the violent plunging the lead



Zinc-treated lead, with copper.  
× 26.

FIG. 4.

receives after the copper is added, and not to the presence of copper at all.

**Conclusions.**—Summing up what has been said, the following points emerge:—

Wastage and destruction of lead in chamber work arise from faults which may readily creep in during the course of acid production, and it is for the makers of sulphuric acid to reduce these chances to the minimum.

Equally, trouble will arise with lead owing to the treatment it may have received at the rolling mills, and it falls on the lead makers to consider carefully this, and to disabuse their minds of the belief that notwithstanding the treatment lead may receive it will always anneal and recover at ordinary temperatures.



Pure Pattinsonian lead.  
× 26.

FIG. 5.

Analysis alone is of no value in disclosing the source of trouble in lead, but if used in conjunction with micrographic work progress would be made.

I am satisfied that there is no alloy of copper with lead at the end of the scale where lead predominates, or in other words, it is not possible to produce a solid solution of copper in lead.

The vagaries as between the results obtained with Parkes and Pattinsonian lead which have been treated with copper lead to the conclusion that the protective value of the copper is mythical. There might be some value in treating the lead at the last stage of refining with a proportion of liquid copper, which, after thorough stirring through the molten charge, should be allowed to settle off. It should not be poured along with the lead. In this case the copper might act beneficially as a scour by alloying



Zinc-treated lead.

x 20.  
FIG. 6.

itself with certain prejudicial metals which are likely to be present with the lead, but great care would be required to see that all the copper was actually settled out, otherwise in the atmosphere of an acid chamber there is every probability that destructive galvanic action would be set up by the presence of the copper.



Pattinsonian lead, with copper.

x 20.  
FIG. 7.

Experience with plant actually built with copper-treated lead has so far shown it to have a shorter lifetime than a similar quality of untreated lead.

All our experiments and observations, so far, still incline us to favour the use of Pattinsonian lead against Parkes, and as zinc is insoluble in lead, the presence of any zinc accidentally left in after separation of silver would be quite as detrimental as that observed in the case of copper, with this difference, that, owing to the copper being readily seen, it is much easier to see when mischief has been caused by that metal than it would be in the case of zinc, which cannot be so readily detected by micro-examination.

## Communications.

### THE RELATION BETWEEN THE DENSITY AND COMPOSITION OF GLASSES.

BY W. L. BAILLIE.

Prior to the researches of Winkelmann and Schott (Winkelmann, *Ann. Phys. Chem.*, 1833, 49, 401; 1897, 67, 105; Winkelmann and Schott, *loc. cit.*, 1894, 51, 697, 731) little appears to have been definitely known regarding the relations existing between the composition of glasses and their physical properties. It was shown (*loc. cit.*) from the study of a large number of glasses that many physical constants admit of computation with tolerable accuracy by means of appropriate numerical factors and that, in particular, density may be so calculated. For the majority of the silicate and borosilicate glasses studied, the calculated and observed densities agreed to within 2% or thereby. Larger discrepancies, however, were encountered in the case of zinc borate and lead borate glasses, the greatest being just over 10%.

The relation between density and composition was investigated later by Tillotson (*J. Ind. Eng. Chem.*, 1911, 3, 897; 1912, 4, 820), who proposed considerable modifications in certain of Winkelmann's factors. It was shown from experimental meltings and from data collated from Larsen (*Amer. J. Sci.*, 1909, 28, 163), Wallace (*Z. anorg. Chem.*, 1909, 63, 1), Day and Allen (*Bull. 31, Carnegie Inst. of Washington*) and others, that the new factors so obtained led to distinctly better agreement between calculated and observed values than did the old. Unfortunately, however, Tillotson did not derive factors for  $PbO$  (†) and  $K_2O$ , two important constituents of many varieties of optical glass. In this respect, therefore, his results are incomplete, and full comparison with other systems of density moduli is not directly possible.

In connexion with the work of the Directorate of Chemical Inspection it became necessary to ascertain the applicability of published moduli to certain types of glassware. Winkelmann's values being found to lead to greater discrepancies between calculated and observed densities than was desirable, an endeavour was made initially to complete Tillotson's series of factors. Consideration of his data, however, led to a distinctly lower value for the density of combined  $SiO_2$ , and it became apparent that it would be necessary to evaluate new factors on this basis. Conditions rendered it impossible to prepare suitable series of glasses of known and simple composition. The problem had therefore to be attacked by consideration of published data and determination of the densities of available glasses already analysed.

Subsequent to the completion of this work, English and Turner published two papers on the density of lime-soda and magnesia-soda glasses respectively (*J. Soc. Glass Tech.*, 1920, 4, 153, 126). They showed that the original factors of Winkelmann and Schott are inadequate, and that Tillotson's factors, although superior, do not satisfactorily reproduce their experimental results. A few factors are tentatively proposed by them, but final pronouncement is delayed pending further work. It is shown, however, that certain results are at variance with Tillotson's deductions from Larsen's data (*loc. cit.*). This anomaly is discussed below.

It is customary to express the composition of glasses in terms of constituent oxides. The calculation and application of density moduli is based on the assumption that to each of these oxides, in

\* Adequate summaries of these researches are given, with numerical data, in Hovestadt's "Jena Glass" (Macmillan), 1902, pp. 144 et seq.

† See note to Table 11.

its combined state, may be assigned a constant, though empirical "density."

Let the analytical results for a glass be  $a\%$ ,  $b\%$ ,  $c\%$ , .....  $n\%$  of the  $n$  oxides composing it, and let the sum of the determined constituents be  $S$ —a quantity which should differ only slightly from 100. If, now, these oxides have, in their combined states, densities  $A$ ,  $B$ ,  $C$  .....  $N$ , and if the density of the glass, as determined by experiment, be  $X$ , then, equating the sum of the partial volumes to the total volume, we have:—

$$\frac{a}{A} + \frac{b}{B} + \frac{c}{C} + \dots + \frac{n}{N} = \frac{S}{X} \dots \dots \dots (1)$$

Given a suitable series of  $n$  glasses of identical qualitative composition, numerical values may be obtained for the  $n$  density moduli,  $A$ ,  $B$ ,  $C$  .....  $N$  from the  $n$  simultaneous equations which are derived. Satisfactory values having been obtained for the commoner constituents, it is comparatively simple to extend the series to include other oxides. The concordance shown below between calculated and observed results is substantial indication of the validity of the basic assumption, but the anomaly mentioned above would appear to indicate that the nature of the remaining oxides may not on occasion be without effect on the density moduli of certain constituents.

Generally, by analogy with liquid mixtures, it is to be expected that the densities calculated for the combined oxides will exceed those observed for the same oxides in the free state. This was stated to hold without exception by Winkelmann and Schott (*loc. cit.*). Tillotson, however, found that  $Al_2O_3$  appears to expand in combination with resulting diminution in density. This observation has been confirmed, and it is shown below that  $Sb_2O_3$  behaves similarly. At this stage it is not possible to express a definite opinion on the significance of these conclusions, but it is suggested that combination of these oxides with silica and bases to form vitreous bodies may be attended with fission of their molecules. The following table shows the difference between the densities of the free and combined oxides, as found by the observers named. In the last column are given the moduli now proposed.

TABLE I.  
Densities of glass-forming oxides in free and combined states.

Oxide.	Density in free state.		Density in combined state.		
	Determined.	Remarks.	Winkelmann.	Tillotson.	Proposed.
$SiO_2$	2.22	Dewille 1855	2.3	2.3	2.24
$Al_2O_3$	3.85	Rammelsberg	4.1	2.75	2.75
$Sb_2O_3$	6.69	—	—	—	3.00
$B_2O_3$	1.79	Clarke	1.9	2.24	2.20
$As_2O_3$	3.74	—	4.1	(4.1)	3.33
$ZnO$	5.65	Schröder	5.9	5.9	5.94
$BaO$	5.00	Clarke	7.0	7.0	7.20
$CaO$	3.80	Molassan	3.3	4.1	4.30
$MgO$	3.60	Clarke & Moissan	3.8	4.0	3.25 <sup>1</sup>
$PbO$	9.80	Clarke	9.6	9.6	10.80
$K_2O$	2.66	—	2.8	(2.8)	3.20
$Na_2O$	2.55	—	2.6	2.8	3.20

<sup>1</sup> Provisional only.

<sup>2</sup> In absence of alkali oxides, the value 4.30 is to be taken.

The analogy with liquid mixtures, which is not invalidated by the behaviour of  $Al_2O_3$  and  $Sb_2O_3$ , explains simply the considerable discrepancies recorded by Winkelmann and Schott in the case of the borate glasses, and also the differences now found in the case of the two exceedingly dense flint glasses (Nos. 8 and 9, Table 9). It will be seen that all the markedly exceptional glasses are sub-

stantially of the nature of simple borates or silicates. The glasses which show close agreement are invariably of the more usual double silicate, borosilicate, or aluminosilicate types. It would therefore appear that this treatment is, in general, inapplicable to glasses of the simplest composition. Fortunately this reservation is generally of little moment since, in practice, such glasses are only very rarely encountered.

It will be clear from the manner of their derivation that all these moduli are interdependent, so that errors in the values of the moduli for the commonest oxides will be reflected in all deductions based thereon. Since silica is the major component by weight in the majority of glasses, and since its modulus is the smallest, it follows that the partial volume of combined silica is generally the largest in a glass. For these reasons attention was primarily directed to the determination of the density of combined silica, employing data from Tillotson (*loc. cit.*). For reasons discussed below, Winkelmann's data were disregarded.

The method of obtaining these moduli renders it possible to achieve absolute concordance between calculated and observed value for any small series of glasses all containing the same oxides and to the same number as there are glasses in the series—e.g., four glasses containing  $SiO_2$ ,  $CaO$ ,  $Na_2O$ , and  $Al_2O_3$  in different proportions. This misleading appearance of accuracy must be sacrificed if good general agreement is to be obtained with glasses of widely different compositions. The moduli now proposed have been derived for this more general purpose. Certain causes are considered below which may impair the degree of correlation which may be obtained when many glasses are in question.

It is known that the thermal history of a glass exercises a profound influence upon its physical and mechanical properties. "Rupert's drops" represent a familiar laboratory example, while the "tempered"—more accurately, case-hardened—glass of de la Bastie represents an attempted commercial utilisation of the resistance to impact and sudden changes in temperature shown by glass which has been subjected to appropriate heat treatment. The presence of strains, due to inefficient annealing, has been shown to augment expansibility, to reduce thermal endurance, and to diminish density. In the case of certain  $MgO$ — $Na_2O$ — $SiO_2$  glasses, English and Turner (*J. Soc. Glass Tech.*, 1920, 4, 153) found that careful annealing of air-cooled rods caused increases in density of as much as 0.01. Similar figures have been recorded for window glass (Chevandier de Valdrôme and Wertheim, *Comptes rend.*, 1845). Zulkowski has made the suggestion that "plaining" yields a homogeneous glass primarily because it allows of the attainment of chemical equilibrium in the mass (*Chem. Ind.*, 1900, 23, 108). If this be accepted it follows that faulty heat-treatment during founding may occasion discrepancies between observed and calculated densities which annealing cannot eliminate.

Tillotson has rightly insisted that the densities attributed to the (combined) constituent oxides of glasses must be regarded as controlled by more than the density of the uncombined oxides alone. The general order of the moduli would appear to indicate that the density of the free oxide is a controlling factor. On the other hand, it is yet to be proved that mode of combination is without effect. Indeed, the anomalous behaviour of  $MgO$ , described later, suggests that this factor is occasionally of considerable importance.

The duplex character of the "plaining" process premised by Zulkowski (*loc. cit.*) leads to the conclusion that during the process some at least of the oxides must alter their state of combination till equilibrium is reached. Up to this point no constant modulus may be applicable. It is conceivable,

indeed, that "plaining" may not entirely correct this, so that, depending on the qualitative and quantitative composition of the glass, application of any rigid modulus may lead to imperfect concordance between calculated and observed results. On the other hand, it appears hardly likely that considerable differences can exist between the moduli appropriate to the various states of combination, so that the differences between calculated and observed results should be, in all probability, of the order of 1–2%. This conclusion is borne out in the case of MgO, the only oxide which, so far, has been found to show any variation in modulus with mode of combination. In the case of experimental melts it is comparatively simple to carry "plaining" to the required degree, and in such cases agreement between calculated and observed results should be close. Optical glasses, on the other hand, do not always admit of such prolonged heating, and for this reason less rigorous agreement may not unreasonably be expected. It is noteworthy, in this connexion, that not only is this state of affairs realised in the data below, but that marked divergencies between calculated and observed densities in the case of glasses of extreme composition are generally negative, i.e., the calculated densities are high.

The effect of permissible analytical errors upon the calculated densities may be shown generally as under:—

Let the true composition of a glass be  $a\%$ ,  $b\%$ ,  $c\%$ ,..... $n\%$  respectively of its  $n$  constituents. Then—

$$\frac{a}{A} + \frac{b}{B} + \frac{c}{C} + \dots + \frac{n}{N} = \frac{100}{X} \quad (2)$$

If, now, the analytical results be in error by amounts  $\alpha$ ,  $\beta$ ,  $\gamma$ ,..... $v$ , then the calculated density,  $X_1$ , is given by

$$\frac{a+\alpha}{A} + \frac{b+\beta}{B} + \frac{c+\gamma}{C} + \dots + \frac{n+v}{N} = \frac{100+a+\beta+\dots+v}{X_1} \quad (3)$$

Rewriting and substituting from (2), we have

$$\frac{100}{X} + \Sigma \left( \frac{\alpha}{A} \right) = \frac{100 + \Sigma(\alpha)}{X_1} \quad (4)$$

Whence:—

$$X_1 = X \left( \frac{100 + \Sigma(\alpha)}{100 + X \Sigma \left( \frac{\alpha}{A} \right)} \right) \quad (5)$$

In general the differences between  $\Sigma(\alpha)$  and  $X \Sigma \left( \frac{\alpha}{A} \right)$  are slight, so that discrepancies due to allowable analytical variations are much less considerable than those mentioned above.

It would therefore appear that experimental results may not be reproduced if the glasses tested be not free from strain or have been insufficiently plained during manufacture. The effect of other ingredients on the moduli of particular oxides is discussed, in the case of MgO, after Table 7. Slight inaccuracies are also possible in the moduli themselves, depending on the operation of the foregoing sources of error upon the glasses examined. Fuller discussion on this point is not at the moment possible, sufficient data being lacking.

It was initially hoped that the wide range of glasses studied by Winkelmann might be employed with advantage in calculating density factors. (Seventy-two glasses in all are described, including silicates, borosilicates, aluminosilicates, borates, phosphates, etc.; vide Hovestadt, "Jena Glass," pp. 146–147.) Consideration of the compositions given, however, led to the conclusion that they were not based upon analysis. The fact that the sum of the constituent oxides always lay between 99.9 and 100.1 was taken as at least an indication of recalculation of results to the 100% basis—if not, indeed, calculation from the batch. The absence of traces of impurity normally found even in optical glasses, due to impurities in the raw materials or to solution of the pot substance, appeared to confirm the latter supposition unless all the melts had been made in platinum, a fact which surely would have been recorded. Confirmation of the final conclusion that the results were calculated from the batch was found in a paper by Zschimmer (Z. Elektrochem., 1905, 11, 629). This finally invalidates Winkelmann's data for the purpose in question, and some of his glasses, selected at random, have shown that even poorer agreement may now be found than with his original moduli,

TABLE 2.  
Composition and densities of Na<sub>2</sub>O–BoO–SiO<sub>2</sub> glasses.

No.	Composition by weight.			Observed density.	Calculated density.			Error (observed—calculated).		
	SiO <sub>2</sub> .	Na <sub>2</sub> O.	BoO.		Winkelmann.	Tillotson.	Proposed.	Winkelmann.	Tillotson.	Proposed.
1	74.50	25.50	—	2.437	2.37	2.410	2.426	0.067	0.027	0.011
2	66.15	20.85	13.00	2.64	2.59	2.627	2.642	0.050	0.013	–0.002
3	61.70	19.20	19.10	2.752	2.725	2.745	2.762	0.027	0.006	–0.010
4	53.00	18.40	33.60	3.05	3.03	3.065	3.076	0.030	–0.005	–0.016
5	71.70	22.45	5.65	2.491	2.45	2.496	2.508	0.041	–0.005	–0.017
6	67.60	19.85	12.55	2.633	2.57	2.614	2.629	0.063	0.019	0.004
7	64.55	16.05	18.47	2.759	2.69	2.721	2.726	0.069	0.033	–0.012
8	61.30	14.50	24.20	2.824	2.80	2.834	2.836	0.024	–0.018	–0.016
9	58.30	12.00	29.70	2.934	2.925	2.932	2.936	0.009	–0.006	0.001
10	55.34	9.50	35.10	3.072	3.05	3.073	3.078	0.017	–0.012	0.010
11	52.45	9.94	40.60	3.212	3.195	3.224	3.229	0.017	0.004	0.029
12	49.95	4.05	46.00	3.367	3.35	3.363	3.368	0.017	0.004	0.029
13	45.91	2.29	50.80	3.476	3.46	3.512	3.483	–0.024	–0.036	–0.007
14	44.25	—	55.75	3.65	3.67	3.676	3.637	–0.020	–0.026	0.013
15	79.09	15.95	5.15	2.45	2.43	2.454	2.443	0.030	0.006	0.017
16	75.85	14.80	9.35	2.514	2.50	2.525	2.512	0.014	–0.011	0.002
17	67.45	12.20	20.35	2.722	2.73	2.733	2.721	0.002	–0.011	0.001
18	62.60	10.80	26.60	2.845	2.84	2.867	2.856	0.005	–0.022	–0.011
19	57.20	9.20	33.60	3.06	3.01	3.034	3.023	0.050	0.026	0.037
20	51.14	7.35	41.50	3.224	3.265	3.245	3.236	–0.041	–0.021	–0.012
21	35.84	2.76	60.40	3.95	3.88	3.991	3.891	0.070	0.049	0.059
Arithmetical mean of errors					..	..	..	0.033	0.018	0.015
Mean square error					..	..	..	0.040	0.023	0.020
Algebraical sum of errors					..	..	..	0.522	0.005	0.114

the discrepancies being occasionally many times greater than in the case of the analysed glasses discussed below. On all grounds, therefore, Winkelmann's series of factors may be discarded as inherently inadequate to reproduce experimental results with any reasonable degree of accuracy.

Examination of the available data indicated that Tillotson had already discussed the most serviceable observations. Comparison of his tables led to the choice of his 21 melts in the  $\text{Na}_2\text{O}$ — $\text{BaO}$ — $\text{SiO}_2$  series from which to derive the factor for combined  $\text{SiO}_2$ . By selection of three sets of three melts, three slightly different values for  $\text{SiO}_2$  were obtained, and from these values for  $\text{Na}_2\text{O}$  and  $\text{BaO}$  were calculated. The densities of the twenty-one glasses were then computed, using each series of factors in turn, and the results led to the adoption of the following mean values:— $\text{SiO}_2$ , 2.24;  $\text{Na}_2\text{O}$ , 3.20;  $\text{BaO}$ , 7.20. Employing these moduli, Table 2 has been constructed.

It will be clear that, however judged, Winkelmann's factors give the poorest results. There is less difference between the remaining systems and correspondingly closer agreement between calculated and observed results. The slight diminution in the mean square error, however, is regarded as indicating that the factors now proposed are slightly superior.

Larsen's data for  $\text{CaO}$ — $\text{MgO}$ — $\text{SiO}_2$  melts were employed for evaluating the moduli for these bases, the above value for  $\text{SiO}_2$  being utilised. As before,

several pairs of factors were deduced and applied to the glasses in turn, when the greatest degree of concordance between calculated and observed densities was found to be obtained using the values  $\text{MgO}$  4.30;  $\text{CaO}$  4.30.

By means of certain of the foregoing values the density of combined  $\text{Al}_2\text{O}_3$  was deduced from the results of Day and Allen for albite-anorthite mixtures (*loc. cit.*). In agreement with Tillotson, the mean value best representing their results is 2.75. It therefore appears that Winkelmann's value, 4.1, is definitely much too high. The factors have been applied also to English and Turner's  $\text{CaO}$ — $\text{Na}_2\text{O}$ — $\text{SiO}_2$  glasses, with results as given.

It will be seen that Winkelmann's factors again fail to reproduce experimental results, and that the factors now proposed show a slight but definite advantage over those published by Tillotson.

Scrutiny of the densities of  $\text{MgO}$ — $\text{Na}_2\text{O}$ — $\text{SiO}_2$  glasses recorded by English and Turner (*loc. cit.*) showed that the modulus for combined  $\text{MgO}$  obtained from Larsen's data was quite unsuitable, since its adoption would necessitate an increase in density on successive replacement of  $\text{Na}_2\text{O}$  by  $\text{MgO}$ , and not a slight fall as was found experimentally. It was found, however, on consideration of the figures, that the value 3.25 for  $\text{MgO}$ , in conjunction with the figures previously adopted, expressed the experimental results very closely (Table 7). The compositions and densities of the glasses are shown in Table 6.

TABLE 3.  
Compositions and densities of  $\text{CaO}$ — $\text{MgO}$ — $\text{SiO}_2$  glasses.

No.	Composition by weight.			Observed density.	Calculated density.			Error (observed—calculated).		
	$\text{CaO}$ .	$\text{MgO}$ .	$\text{SiO}_2$ .		Winkelmann.	Tillotson.	Proposed.	Winkelmann.	Tillotson.	Proposed.
1	48.20	—	51.80	2.904	2.693	2.920	2.913	0.206	-0.016	-0.009
2	45.80	2.01	52.19	2.899	2.700	2.910	2.905	0.199	-0.011	-0.006
3	41.09	5.91	53.00	2.891	2.701	2.897	2.891	0.190	-0.006	—
4	35.70	10.43	53.87	2.881	2.708	2.879	2.875	0.175	-0.002	0.006
5	30.84	14.43	54.73	2.872	2.709	2.863	2.860	0.163	0.009	0.004
6	28.92	16.04	55.04	2.859	2.711	2.855	2.855	0.148	0.004	0.003
7	25.85	18.59	55.56	2.854	2.713	2.848	2.846	0.141	0.005	0.007
8	19.28	24.06	56.66	2.834	2.718	2.829	2.827	0.116	0.006	0.007
9	14.46	28.07	57.47	2.821	2.722	2.815	2.814	0.099	-0.007	-0.006
10	4.82	39.09	56.09	2.730	2.730	2.787	2.786	0.050	-0.003	-0.003
11	2.41	38.10	59.49	2.777	2.733	2.730	2.780	0.044	-0.015	-0.015
12	—	40.10	59.90	2.753	2.735	2.773	2.773	0.023	—	—
Arithmetical mean of errors				..	..	..	..	0.130	0.008	0.007
Mean square error				..	..	..	..	0.143	0.009	0.008
Algebraical sum of errors				..	..	..	..	1.554	-0.023	0.005

TABLE 4.  
Composition of  $\text{CaO}$ — $\text{Na}_2\text{O}$ — $\text{SiO}_2$  glasses.

Percentage composition by weight.								
No.*	$\text{SiO}_2$ .	$\text{Na}_2\text{O}$ .	$\text{CaO}$ .	$\text{MgO}$ .	$\text{Al}_2\text{O}_3$ .	$\text{Fe}_2\text{O}_3$ .	$\text{As}_2\text{O}_3$ †	Total.
1	74.05	25.34	0.21	tr.	0.24	0.14	—	99.98
2	73.92	23.80	1.50	tr.	0.20	0.14	—	99.56
3	74.08	23.00	2.61	tr.	0.21	0.15	—	100.05
4	74.07	21.50	3.81	tr.	0.28	0.14	—	99.80
5	73.78	20.78	4.50	0.15	0.38	0.19	—	100.22
6	73.13	19.38	6.26	0.21	0.53	0.61	—	100.00
7	74.41	17.20	7.45	0.24	0.30	0.40	—	99.81
8	74.99	16.00	8.16	0.26	0.41	0.16	—	100.08
9	74.96	14.88	9.36	0.28	0.42	0.21	—	100.15
10	74.59	14.22	10.38	0.30	0.45	0.17	—	100.49
11	74.93	13.02	11.68	0.31	0.38	0.31	0.30	100.47
12	66.71	12.72	18.17	0.65	1.41	0.25	0.47	100.10
13	69.73	11.22	17.38	0.55	0.50	—	—	—

\* From English and Turner, *loc. cit.*

† Factor assumed, 3.33 (provisional only).



TABLE 5.  
Densities of  $\text{CaO-Na}_2\text{O-SiO}_2$  glasses.

No.	Observed density.	Calculated density.			Error (observed—calculated).		
		Winkelm.	Tillotson.	Proposed.	Winkelm.	Tillotson.	Proposed.
1	2.485	2.383	2.408	2.429	0.052	0.027	0.006
2	2.438	2.383	2.421	2.434	0.055	0.017	0.004
3	2.442	2.389	2.430	2.441	0.053	0.012	0.001
4	2.446	2.391	2.437	2.445	0.055	0.009	0.001
5	2.451	2.401	2.444	2.450	0.050	0.007	0.001
6	2.460	2.419	2.462	2.465	0.041	-0.002	-0.005
7	2.465	2.418	2.463	2.461	0.045	0.002	0.004
8	2.468	2.413	2.464	2.460	0.053	0.004	0.005
9	2.476	2.419	2.474	2.467	0.057	0.002	0.009
10	2.484	2.429	2.481	2.475	0.055	0.003	0.009
11	2.490	2.431	2.492	2.481	0.059	-0.002	0.009
16	2.527	2.523	2.537	2.576	0.064	—	0.011
17	2.555	2.490	2.562	2.554	0.065	-0.007	0.001
Arithmetical mean of errors .. .. .					0.054	0.007	0.005
Mean square error .. .. .					0.055	0.010	0.006
Algebraical sum of errors .. .. .					0.704	0.072	0.059

TABLE 6.  
Compositions of  $\text{MgO-Na}_2\text{O-SiO}_2$  glasses.

No.*	Percentage composition by weight.				
	$\text{SiO}_2$	$\text{Na}_2\text{O}$	$\text{MgO}$	$\text{CaO, Al}_2\text{O}_3$ , etc.	Total.
24	74.07	24.23	1.10	0.37	99.87
25	74.35	23.46	1.85	0.75	100.41
26	75.00	22.12	2.49	0.28	99.97
27	75.16	20.09	3.47	0.46	99.78
28	75.19	19.83	4.20	0.52	99.74
329	70.30	18.26	5.09	0.74	100.39
330	77.09	10.05	6.10	0.68	99.92
331	76.68	15.77	6.87	0.93	100.25
332	76.86	14.55	7.46	0.93	99.80
389	77.77	13.40	8.45	0.56	100.17
390	78.28	11.76	9.30	0.91†	100.25

\* From English and Turner, *loc. cit.*  
† Including 0.5%  $\text{CaO}$ .

Details of minor ingredients are not given in the original. A mean modulus 3.0 has been assigned for use with the factors now proposed, and 3.5 for use with Winkelmann's factors.

The factors tentatively proposed by English and Turner are as follows:—

	Series 1.		Series 2.	
$\text{SiO}_2$ .. .. .	2.20	2.30	2.30	2.30
$\text{Na}_2\text{O}$ .. .. .	3.47	2.9	2.9	2.9
$\text{MgO}$ .. .. .	3.38	2.9	2.9	2.9

TABLE 7.  
Densities of  $\text{MgO-Na}_2\text{O-SiO}_2$  glasses.

No.	Observed density.	Calculated density.					Error (observed—calculated).				
		Winkelm.	Tillotson.	Turner, Series 1.	Turner, Series 2.	Proposed.	Winkelm.	Tillotson.	Turner, Series 1.	Turner, Series 2.	Proposed.
24	2.482	2.383	2.394	2.440	2.443	2.430	0.049	0.038	-0.008	0.011	0.002
25	2.430	2.388	2.394	2.433	2.436	2.429	0.042	0.036	-0.003	0.006	0.001
26	2.427	2.389	2.397	2.427	2.433	2.434	0.058	0.030	0.006	0.003	0.003
27	2.424	2.394	2.412	2.431	2.438	2.429	0.030	0.012	-0.007	0.014	0.004
28	2.421	2.399	2.418	2.431	2.430	2.419	0.022	0.003	-0.010	0.018	0.002
329	2.415	2.404	2.418	2.413	2.425	2.409	0.011	-0.003	0.002	0.010	0.006
330	2.410	2.409	2.429	2.412	2.429	2.406	0.001	-0.019	-0.002	0.019	0.004
331	2.406	2.417	2.425	2.414	2.430	2.410	-0.011	-0.022	-0.006	0.024	-0.004
332	2.403	2.420	2.447	2.419	2.438	2.407	-0.017	-0.044	-0.016	0.085	-0.004
389	2.307	2.424	2.441	2.397	2.419	2.397	-0.027	-0.044	—	0.022	—
390	2.395	2.440	2.446	2.397	2.427	2.399	-0.045	-0.051	-0.002	0.032	-0.004
Arithmetical mean of errors .. .. .							0.027	0.027	0.005	0.015	0.003
Mean square error .. .. .							0.035	0.035	0.007	0.020	0.003
Algebraical sum of errors .. .. .							0.093	-0.064	-0.054	0.197	0.010

Comparison of Tables 3 and 7 shows that the experimental results are adequately reproduced by the systems of factors proposed, subject to the adoption of the smaller factor for  $\text{MgO}$  in the latter case. The density of combined  $\text{MgO}$  therefore appears to exceed that of the free  $\text{MgO}$  in the  $\text{CaO-MgO-SiO}_2$  glasses and to be less in the  $\text{Na}_2\text{O-MgO-SiO}_2$  glasses. In the former series of melts it is reasonable to suppose that  $\text{CaSiO}_3$  and  $\text{MgSiO}_3$  are formed and that the metasilicates are mutually soluble. Indeed, it may simply be demonstrated graphically or by calculation that this assumption meets the facts. On the other hand, it is established from the properties of  $\text{Na}_2\text{O-MgO-SiO}_2$  melts that they could not have resulted simply in mutual solution of silicates of sodium and magnesium, but that further reactions occurred with the formation of more stable compounds possessing in increasing degrees those qualities of chemical durability usually associated with the term "glass" as the magnesia content increased. The conclusion appears to be indicated that, in alkali-containing melts, the density of combined  $\text{MgO}$  is less than that of the free oxide, thus making it comparable with  $\text{Al}_2\text{O}_3$  and  $\text{Sb}_2\text{O}_3$ , but that an increase in density occurs in melts of such composition that no chemical action is probable beyond the formation of a simple magnesium silicate. For this reason, therefore, the value 3.25 has been adopted for the density of combined  $\text{MgO}$  in ordinary glasses, and the results tabulated below have been recalculated

(from the value 4.30 previously adopted) on this basis.

None of the remaining constituents of optical glasses being included in the available published data, the list of moduli was extended from 34 glasses of known composition. Density determinations were made by means of a specific gravity bottle. This was chosen primarily for convenience and immediate availability and the fact that, besides involving neither unusual nor elaborate accessories, it admits of the attainment of a sufficient degree of accuracy without unduly laborious supervision. It is unnecessary in this work to carry results beyond the fourth decimal place to secure accuracy in the third, and the method adopted was found, by the concordance between duplicate determinations, to be sufficiently delicate. This degree of accuracy corresponds to

an error rarely exceeding 0.04% and further refinement is needless. Densities were determined on clean, freshly-broken fragments which were brushed free from dust, thus almost wholly eliminating trouble from adherent air-bells. Water was employed throughout, none of the glasses exhibiting sufficient solubility to negative its use. The succeeding tables show the compositions and densities of the glasses employed, and the degree of correlation between the observed values and those calculated by the various systems of factors.

Density factors for PbO and K<sub>2</sub>O were calculated to be 10.30 and 3.20 respectively. After making correction for the small amounts of Sb<sub>2</sub>O<sub>3</sub>, ZnO, and B<sub>2</sub>O<sub>3</sub> by means of factors subsequently deduced, the following densities were calculated.

The factor 5.94 was deduced for ZnO from No. 1 of Table 10 and the small corrections for minor con-

TABLE 8.  
*Composition of various lead glasses.*

No.	Percentage composition by weight.									
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub> *	B <sub>2</sub> O <sub>3</sub>	Sb <sub>2</sub> O <sub>3</sub> †	PbO.	ZnO.	MgO.	K <sub>2</sub> O.	Na <sub>2</sub> O.	Total.
1	45.15	0.93	—	0.21	46.47	—	—	1.04	6.43	100.23
2	47.00	1.26	—	0.19	44.44	—	—	0.24	7.00	100.13
3	45.35	1.00	—	0.20	46.03	—	—	0.54	8.89	100.01
4	40.41	0.68	—	0.51	49.83	—	—	7.25	1.17	99.85
5	39.73	0.62	—	0.28	50.73	—	0.13	6.98	1.53	100.25
6	53.39	0.72	—	0.22	35.79	—	—	0.06	0.88	100.06
7	53.12	0.51	—	0.35	35.93	—	—	8.01	1.40	99.92
8	19.90	0.87	—	0.54	76.69	0.22	0.22	0.81	0.92	100.17
9	19.80	0.64	0.22	0.35	77.86	—	—	0.27	1.72	100.41

\* In most cases the glasses contained a small proportion of Fe<sub>2</sub>O<sub>3</sub>, but it was considered unnecessary to separate this. The figures given are, therefore, inclusive.

† Including traces of CuO, derived from the sleeves employed in preparing samples for analysis.

TABLE 9.  
*Densities of lead glasses.*

No.	Observed density.	Calculated density.			Error (observed—calculated).		
		Winkelmänn.	Tillotson.	Proposed.	Winkelmänn.	Tillotson.	Proposed.
1	3.669	3.632	3.641	3.657	0.037	0.028	0.012
2	3.559	3.552	3.553	3.566	0.007	0.006	0.007
3	3.638	3.617	3.631	3.641	0.021	0.007	0.003
4	3.878	3.828	3.798	3.867	0.050	0.020	0.011
5	3.868	3.861	3.853	3.891	0.007	0.015	0.023
6	3.257	3.257	3.245	3.254	—	0.012	0.003
7	3.251	3.259	3.256	3.262	0.008	0.005	0.011
8	5.889	5.652	5.820	5.736	0.237	0.269	0.153
9	5.980	5.686	5.677	5.803	0.294	0.303	0.177
		Arithmetical mean of errors ..	..	..	0.073	0.074	0.044
		Mean square error ..	..	..	0.128	0.135	0.001
		Algebraical sum of errors ..	..	..	0.645	0.615	0.312

TABLE 10.  
*Composition of light flint and zinc crown glasses.*

No.	Percentage composition by weight.										
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Sb <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	ZnO.	BaO.	CaO.	MgO.	PbO.	K <sub>2</sub> O.	Na <sub>2</sub> O.
1	69.60	1.15	0.39	0.23	16.03	—	—	—	—	1.74	10.23
2	60.08	1.07	0.08	—	0.24	2.03	—	0.28	21.95	12.17	1.81
3	61.51	0.56	0.23	—	—	1.58	0.99	—	22.10	12.47	1.21
4	60.74	0.20	0.03	—	1.42	0.20	2.08	0.04	26.67	5.97	2.71
5	61.95	0.10	0.02	—	0.07	1.96	0.55	0.11	24.20	7.75	2.65
6	68.24	1.45	—	—	2.16	—	0.57	0.36	12.00	4.53	13.58
7	67.98	0.71	0.80	0.17	0.20	—	4.35	0.07	6.23	13.56	1.84
8	56.73	0.75	0.83	—	4.73	13.35	—	—	10.38	13.08	1.06
9	53.85	0.56	0.40	0.13	4.21	14.81	—	—	11.47	12.62	1.86

TABLE 11.  
Densities of light flint and zinc crown glasses.

No.	Observed density.	Calculated density.			Error (observed—calculated).		
		Winkelmänn.	Tillotson.	Proposed.	Winkelmänn.	Tillotson.	Proposed.
1	2.606	2.612	2.624*	2.603	-0.006	-0.018	0.003
2	2.880	2.914	2.908	2.908	-0.028	-0.022	-0.022
3	2.885	2.902	2.904	2.903	-0.017	-0.019	-0.023
4	2.885	3.002	3.018	2.996	-0.117	-0.133	-0.110
5	2.907	2.940	2.950	2.938	-0.073	-0.083	-0.071
6	2.715	2.668	2.685	2.693	0.046	0.028	0.020
7	2.609	2.559	2.574	2.603	-0.050	-0.065	-0.094
8	2.979	2.971	2.966	2.972	0.003	0.013	0.007
9	2.982	3.024	3.024	3.035	-0.042	-0.042	-0.053
Arithmetical mean of errors .. .. .					0.043	0.047	0.046
Mean square error .. .. .					0.054	0.060	0.053
Algebraical sum of errors .. .. .					-0.280	-0.341	-0.358

\* The value 9.6 was assumed for PbO since Tillotson appears to have employed this in certain comparisons with Winkelmänn's results (*J. Ind. Eng. Chem.*, 1912, 4, 820) and 2.8 for K<sub>2</sub>O was found, after trial of other factors, to give the best results.

TABLE 12.  
Compositions of crown, borosilicate, and telescopic flint glasses.

No.	Percentage composition by weight.											
	SiO <sub>2</sub> .	Al <sub>2</sub> O <sub>3</sub> .	Sb <sub>2</sub> O <sub>3</sub> .	B <sub>2</sub> O <sub>3</sub> .	ZnO.	BaO.	CaO.	MgO.	PbO.	K <sub>2</sub> O.	Na <sub>2</sub> O.	Total.
1	55.58	0.26	0.30	5.03	—	10.12	—	—	16.48	8.61	4.28	100.64
2	53.67	2.50	20.85	15.08	—	—	—	—	—	4.62	2.02	99.64
3	43.43	2.25	0.50	4.33	7.31	31.39	—	0.50	—	5.10	5.39	100.27
4	40.74	5.95	0.20	4.31	8.51	30.45	—	—	—	3.23	1.24	100.63
5	37.37	4.82	0.30	7.33	7.18	42.34	—	—	—	0.15	0.35	99.84
6	35.81	8.16	0.32	15.83	—	30.80	—	—	—	0.10	0.30	100.32
7	35.08	4.80	0.30	8.30	0.76	44.52	—	—	—	—	0.42	100.24
8	30.41	5.34	0.28	7.50	0.86	43.79	—	—	—	—	0.43	100.61
9	70.45	1.01	0.19	—	—	—	10.47	0.36	—	10.25	7.50	100.32
10	70.40	1.14	0.10	—	—	—	11.93	—	—	0.42	7.52	100.51
11	68.95	1.16	0.43	—	—	—	11.03	0.14	—	17.60	0.78	100.39
12	72.88	1.04	0.42	—	—	—	12.90	—	—	0.20	13.15	100.11
13	72.27	1.63	0.24	1.19	—	—	10.30	—	—	1.75	13.58	100.78
14	69.01	0.94	0.09	5.91	—	—	10.30	—	—	19.30	1.70	100.17
15	70.93	0.73	0.20	5.11	—	—	2.95	—	—	18.80	1.51	100.33
16	70.69	1.19	0.22	—	—	—	6.43	0.17	3.01	4.07	14.31	100.09

TABLE 13.  
Densities of crown, borosilicate, and telescopic flint glasses.

No.	Observed density.	Calculated density.			Error (observed—calculated).		
		Winkelmänn.	Tillotson.	Proposed.	Winkelmänn.	Tillotson.	Proposed.
1	3.024	2.889	2.931	2.980	0.135	0.093	0.044
2	2.535	2.607	2.571	2.532	0.028	-0.036	0.003
3	3.197	3.174	3.197	3.246	0.023	-0.049	-0.049
4	3.208	3.193	3.150	3.180	0.015	0.049	0.028
5	3.567	3.465	3.463	3.527	0.102	0.099	0.040
6	3.283	3.154	3.183	3.325	0.129	0.100	-0.058
7	3.610	3.512	3.523	3.600	0.098	0.087	0.010
8	3.596	3.502	3.493	3.563	0.093	0.084	0.024
9	2.484	2.481	2.504	2.491	0.023	-0.020	-0.007
10	2.489	2.465	2.512	2.497	0.024	-0.023	-0.008
11	2.468	2.483	2.516	2.528	0.015	-0.048	-0.055
12	2.494	2.444	2.502	2.493	-0.050	-0.008	0.001
13	2.503	2.434	2.487	2.490	0.069	0.010	0.013
14	2.465	2.393	2.426	2.472	0.062	0.029	-0.017
15	2.453	2.390	2.421	2.458	0.063	0.032	0.005
16	2.532	2.481	2.522	2.525	0.051	0.010	0.007
Arithmetical mean of errors .. .. .					0.061	0.046	0.023
Mean square error .. .. .					0.072	0.058	0.030
Algebraical sum of errors .. .. .					0.871	0.483	-0.029

stituents have been applied from the factors calculated later.

Employing the factors 2.90 and 3.00 for  $B_2O_3$  and  $Sb_2O_3$  respectively, the following were calculated.

The following table summarises the errors found in applying the various systems of moduli to the glasses discussed.

TABLE 14.  
Summary of errors with 91 glasses.

	Winkelmann.	Tillotson.	Proposed.
Arithmetical mean of errors	0.058	0.029	0.018
Mean square error .. ..	0.071	0.041	0.026
Algebraical sum of errors ..	4.109	0.729	0.118

#### Summary.

(1) The relation between the density and the composition of glasses has been re-investigated.

(2) The moduli proposed by Winkelmann and Schott have been found to lead to discrepancies exceeding 2% in the case of glasses of normal types. The more satisfactory moduli proposed by Tillotson have been shown to be susceptible of certain improvements.

(3) New moduli are proposed whereby glasses of extreme composition may be included with as high a degree of accuracy as was formerly possible for glasses of normal composition.

(4) Applied to 91 glasses, the new moduli have been found to lead to the smallest average error, the smallest mean-square error, and the lowest algebraical sum of errors.

(5) The theory has been discussed and the reasons for certain discrepancies are indicated.

I wish to acknowledge the assistance of my former colleague, Mr. W. T. Lockett, in the experimental work and the calculations involved. Thanks are due also to Mr. G. H. Perry, O.B.E., Director of Chemical Inspection, for the interest he has shown in this work, and to the Director of Artillery, War Office, for permission to embody in the paper analytical and other results obtained in the work of this laboratory.

Glass Laboratory,  
Directorate of Chemical Inspection,  
Royal Arsenal, Woolwich.

### THE BACTERIAL PURIFICATION OF TRADE WASTES.

BY DR. O. MCGOWAN.

The Ninth Report of the Royal Commission on Sewage Disposal<sup>1</sup> deals with the disposal of liquid wastes from manufacturing processes. The outbreak of the war, however, cut short the study of the subject, and various points requiring investigation were of necessity left untouched or unfinished. One of these was to acquire accurate experimental data with regard to the amenability of different trade wastes to bacterial purification or, in other words, to ascertain the respective volumes which could be treated on a cubic yard of contact bed or percolating filter. Since that time the activated sludge process of sewage purification has gained increasing prominence, and as this process largely conserves the nitrogen of sewage, it may be expected to become more widely applied as time goes on. Still, the data to be given in the present paper will apply so long as the purification of sewage on contact beds and percolating filters continues; and, besides, the relative rates at which different trade effluents undergo oxidation affect the activated sludge process also.

<sup>1</sup> Cd. 7819 (1915).

The bacterial treatment of domestic sewage is now fairly well systematised, the volume which can be dealt with on unit of filter depending on the strength of the sewage or tank liquor. This "strength" is measured in terms of the oxygen required to oxidise fully the organic matter and ammonia present. 100,000 parts by weight of a sewage of average strength require about 100 parts by weight of oxygen for this purpose.<sup>†</sup>

To estimate the strength of a sewage or other polluting organic liquor, a small measured volume is mixed with a large measured volume of good tap water, i.e., with excess of dissolved oxygen, and the oxygen in solution is determined (a) at the time of mixing and (b) after all the oxidisable matter present has been oxidised. The difference between the two figures found gives the amount of oxygen taken up by the sewage or other liquor.

The procedure followed in this determination of dissolved oxygen absorption is to be found in the Eighth Report of the Royal Commission, Vol. II., Appendix, p. 93 *et seq.* [Cd. 6943 (1913)], but a short outline of it may be given here. The requisite dilution having been made with water at a temperature of about 18° C., the liquid is poured quietly, in alternate sections, into two bottles, (a) a smaller one of about 350 c.c. capacity, in which the determination of the initial oxygen is immediately made, and (b) a quarter-Winchester of 600–700 c.c. capacity, with a well-fitting stopper. Both bottles are of course filled full to overflowing. After the stopper of the quarter-Winchester has been firmly inserted and tied down with tape, a small piece of wide rubber tubing of about 20 mm. internal diameter and 50 mm. long is slipped over it, the top of the rubber remaining about 15 mm. above the head of the stopper. The rubber joint is then nearly filled with mercury and the bottle set aside until the oxidation has completed itself. Unless the glass stopper should have been a bad fit, which rarely happens, this forms a satisfactory joint for the purpose.

At the end of the period allowed for this "long aeration," the small quantity of gas which has collected at the top of the bottle is transferred and analysed, the figure for the oxygen which it contains being added to that for the oxygen in the residual liquid. The method of the transference is described in the Eighth Report, Vol. II., Appendix, p. 149. It should, however, be added that bottles which have been jointed for a number of years, like those referred to in this paper, may have the stopper so firmly cemented in that this treatment is at first insufficient to loosen it. In that case, after pouring a drop or two of water on to the stopper, the rubber and mercury (not the tape) should be replaced and another trial at opening made next day. I have in the most persistent cases had to try three or four times in this way before succeeding in getting the stopper loosened. The precautions necessary for the estimation of the residual oxygen in the liquid are detailed in the Eighth Report, p. 149; I might add here that, instead of liberating the iodine in the water in the presence of some mercury and immediately pouring off, an alternative method is to siphon the liquid into a smaller bottle, previously filled with carbon dioxide, rejecting the first and last portions.

The various samples of trade effluent dealt with in this paper were typical ones drawn by the Officers of the Commission between 1907 and 1914, together with a few drawn later. They were fully analysed at the time by Messrs. A. C. Carter, F.I.C., A. F. Girvan, B.Sc., A.I.C., and the late W. G. Winterson, B.Sc., these analyses including

<sup>†</sup> Cf. Fifth Report of the Royal Commission on Sewage Disposal, Appendix IV., p. 1 *et seq.*; also p. 52 *et seq.* [Cd. 4282 (1910)].

the determination of dissolved oxygen absorption for the shorter periods up to five days or so, and of the initial oxygen of the "long aerations." The residual oxygen of the latter and of the separated gases have been estimated recently by myself (in 1920). The actual quantities of these gases (reduced to N.T.P.) in 58 estimations were:—

	Extremes.	Average.
Carbon dioxide ..	0 to 0.07 c.c.	0.02 to 0.03 c.c.
Oxygen ..	0 to 0.65 c.c.	0.27 c.c.
Nitrogen ..	0.46 to 3.15 c.c.	1.78 c.c.

As it may be useful, for purposes of future reference, that the general character of the samples examined should be known, the reference is given to the pages of the Ninth and Sixth Reports of the Commission where, with a few exceptions, the detailed analyses are to be found. The two samples from the jam factories were examined after the publication of these reports, and are therefore not included in them; nor are some of the malt distillery samples. The general character of the latter can, however, be deduced from the other similar samples. Meantime, it will suffice to give here the figure for dissolved oxygen taken up in five days and that required for complete oxidation.

The ratio of the figure for dissolved oxygen absorption in five days at 18° C. to that for complete oxidation is in the case of domestic sewage something like 1:3. A higher ratio—say, 1:6—means that the oxidation as a whole proceeds more slowly, i.e., that the liquor in question is more difficult of oxidation than sewage. Failing, therefore, a full series of estimations for each sample, giving the figures for dissolved oxygen absorption in 2, 5, .....x days, the ratio between the figures obtained for five days and for complete oxidation may in the meantime be taken as a guide for getting at the amenability of the trade liquor to bacterial treatment.

The length of time which should be allowed for the "long aeration" of trade effluents, i.e., for their (practically) complete oxidation in the laboratory, is difficult to state precisely. Probably three months at a temperature of about 18° C. would be sufficient in nearly all cases, but it would be safer to allow six months, either at 18° C. or at laboratory temperature.

Nature of sample.	Reference in 9th Report of Sewage Commission.	A. Dissolved oxygen taken up in 5 days at 18° C.	B. Oxygen required for complete oxidation.	Ratio A : B.	Dilution with tap water for "Long aeration."
-------------------	---	--	--	--------------	--

#### Parts per 100,000 by weight.

<i>Cotton dyeing and printing.</i>					
Final effluent	p. 24	5.9	27.3*	1:4.6	1 in 80
Crude waste.	" 33	11.3	38.4	1:3.4	" 150
Settled efflu't	" 34	7.1	29.4	1:4.1	" 80
Tank liquor.	" 36	13.1	47.6	1:3.6	" 100
Filter effluent	" 36	10.6	44.6	1:4.2	" 100
Final effluent	" 31	27.1	110.0	1:4.1	" 150
Effluent ..	" 26	1.84	8.2	1:4.5	" 25

#### *Cotton bleaching.*

Settled liquor (i.e., final effluent)	p. 43	47.8+x	172.4	?	1 in 400
Final effluent	" 43	50.1	Probably 180 to 190 185.6 Rather below the truth.	1:3.7	" 400
Effluent as discharged	" 47	29.4	No doubt hypo-chlorite in the residual liquid.	?	" 200

#### *Paper mills.*

Exhaust tank liquor	p. 81a	9.8	43.6	1:4.4	1 in 150
Percolating filter effluent	" 84a	8.4	39.3	1:4.7	" 150
Final effluent	" 88	0.73	7.7	1:10.5	" 20

#### *Tanning and fell-mongering.*

Waste liquor from chrome tannery (siphoned from most of its solids)	—	90.5	448.2	1:5.0	1 in 634
Final effluent	p. 133	29.9	(No doubt hypo-chlorite)	?	" 300
Effluent ..	" 125	15.0	65.7	1:4.4	" 300

#### *Jam factories.*

"Sewage" (siphoned)	—	(a) 233.5 (b) 150.6	?	—	1 in 443
Tank liquor..	—	39.8	471.9	1:11.9	" 904

#### *Malt and brewery liquors.*

Steep water..	p. 116	33.4	119.0	1:3.1	1 in 1,000
Secondary bed effluent	" 114	17.6+x	60.3	1: less than 3	" 150

#### *Grain distillery liquors.*

Steep water..	Ref. in 6th Rep. p. 78	28.8†	(a) 103.5 (b) 98.3	1:3.6 1:3.4	1 in 750 " 750
Spent waste ..	" 73	1,940	(a) 5,480 (b) 6,300	1:3.8 1:3.2	" 10,000 " 10,000
Spent waste .. do. (paper filtered)	" 73	850.4	2,443	1:2.9	" 7,000
Spent wash..	" 78	431.6	1,509	1:3.5	" 3,500
Spent wash.. do. (paper filtered)	" 78	1,218	(a) 2,004 (b) 2,374	1:2.5 1:2.4	" 6,000 " 9,000
Spent wash.. do. (paper filtered)	" 73	594†	Not done	?	" 3,500

#### *Malt distillery liquors.*

Pot ale ..	p. 65	2,500	(a) 5,850 (b) 7,335	1:2.3 1:2.9	1 in 15,000 " 15,000
Strong steep liquor	" 65	151 5-7 of 7 days	649†	1:4.2	" 2,000
Second steep liquor	" 65	104 5-7 of 7 days	324†	1:3.1	" 1,000
Mixed steep liquor	" 65	133 5-6 of 6 days	(a) 347 (b) 339	1:2.3	" 1,000 " 1,000
Mixed steep liquor	?	275 5-4 of 4 days	(a) 608 (b) 650	1:2.3	" 1,500 " 1,500
Lined steep liquor	?	135 5-4 of 4 days	(a) 425 (b) 394	1:2.6	" 1,000 " 1,000
Lined steep liquor	?	233 5-4 of 4 days	(a) 543 (b) 521	1:2.3	" 1,500 " 1,500
Lined liquor as going on to filter	?	216 6 days	239†	?	" 600

#### *Spent lees.*

Spent lees ..	—	1.75	(a) 114.3 (b) 121.3	1:67	1 in 250 " 250
Distillate of 100 c.c. from 200 c.c. spent lees	—	—	42.7+x	—	" 50
Distillation residue made up to original volume of 200 c.c.	—	0.61	(a) 83.5 (b) 58.5	1:137 1:86*	" 250 " 250
Spent lees ..	p. 65	4.85	(a) 117.3 (b) 124.3	1:26	" 250 " 250

\* This figure is rather too low.

† 5-6 of 6 days.

‡ 363 in 36 days (1 in 1,000).

§ 243 in 36 days (1 in 500). 295 to 77 days (1 in 1,000).

¶ 202 in 103 days (1 in 500).

† "Disposal of Liquid Refuse from Distilleries" (1909), Cd. 4511.

**Cotton dyeing and printing.**—In the case of the seven samples examined the ratio of A:B varied from 1:3.4 to 1:4.6, the average being 1:4.1—say, 1:4. A waste liquor or partially purified effluent from cotton dyeing and printing, therefore, which takes up about 25 parts of dissolved oxygen from water in five days at 18° C., may be regarded for purposes of bacterial purification as approximately equivalent to a sewage of strength 100.

It is to be noted here that the two lowest ratios, 1:3.4 and 1:3.6, are given by a crude waste and a tank liquor which contained respectively 11.6 and 6.6 parts of suspended organic solids. The foregoing tentative conclusion, therefore, to look upon such waste giving a five days' figure of 25 as equivalent to sewage with a corresponding figure of 33 receives from this some corroboration.

It has also to be borne in mind that here, as in the case of other types of trade effluent, bacterial oxidation will proceed more rapidly the larger the proportion of domestic sewage there is in the mixture.

**Cotton bleaching.**—The data here are too meagre for any conclusions to be drawn, but the probabilities are that effluents of this class would not differ materially from cotton dyeing and printing effluents, provided that no large excess of hypochlorite was left in them. Any large excess of the latter would of course retard bacterial oxidation.

**Paper mills.**—Here, again, the data are very few, but the ratio of the five days' figure to that for complete oxidation may with some degree of probability be taken in the meantime as 1:4.5. This would make a paper mill liquor or effluent with a five days' figure of about 22 equivalent to a sewage with a figure of 33.

**Tanning and fellingmongering.**—The ratio here may also be taken tentatively at 1:4.5.

**Jam factories.**—With almost no data to go upon, nothing can be said here excepting that such liquors are organically very strong and that they are also slow of oxidation. Possibly the proportion of nitrogen to carbon may be too small for the best rate; in these two samples the combined nitrogen would amount to only about 2 and 3 parts per 100,000, respectively. Pending further results, I think that the ratio A:B might be taken at about 1:10.

**Malt and brewing liquors.**—Although there are only two results here, they indicate that these liquors are readily oxidisable, the ratio A:B being much the same as that for domestic sewage.

**Distillery waste.**—The purification of waste distillery liquors is gone into in detail in the Sixth Report of the Commission. The samples cited in the tables were drawn for the purposes of that report in 1908-9 (one in 1907).

**Grain distilleries.**—Here we have four samples altogether of steep water, spent waste, and spent wash (apart from the paper-filtered samples), and three of them were aerated in duplicate. The four gave a ratio of A:B=1:2.5 to 1:3.6, average 1:3.0. Such liquors, therefore, with a five days' figure of about 33 and a strength of 100 would be equivalent to a sewage of strength 100. The actual strength of the undiluted liquors is of course excessive—in the above samples reaching as high as 50 to 60 times the strength of an average sewage. Considering the very high dilutions (up to 1 in 10,000) that had to be made here, the agreement between the figures for dissolved oxygen absorption by the duplicate samples was fairly good. In the second sample of spent waste, which was examined both before and after filtration through paper, 1 part of the suspended solids present required 1.6 parts of oxygen for oxidation, while 1 part of the soluble solids required 4.0 parts oxygen.

**Malt distilleries.**—The results (leaving spent lees for the moment out of account) are similar to those given by grain distillery liquors. The ratio A:B in

the first seven samples varies from 1:2.3 to 1:4.2, average 1:2.8. As modifying this, however, it must be noted that the figures taken for the five days' dissolved oxygen absorption are not quite the true figures; the true figure would probably be more than that for five-sevenths of seven days, while it might be less than that for five-fourths of four days. On the whole, therefore, the ratio may safely be taken as about 1:3—the same as for domestic sewage. The actual strength of the liquors is great, notably that of the pot ale; the others were less strong than those from the grain distillery. With the exception of the pot ale sample, where the very high dilution of 1 in 15,000 had to be made for the long aeration, the other duplicates showed very fair agreement.

**Spent lees.**—The volume of spent lees in a malt distillery, as compared with that of the pot ale and steep liquor, is so small, and its organic strength so much less, that it hardly requires detailed consideration here. The results obtained with the two samples tabulated above are, however, interesting as showing the slow rate at which oxidation went on. Mixed with the other distillery liquors, therefore, the spent lees will tend to retard the bacterial oxidation of the whole, though its relatively small volume renders this of little moment. I cannot account for the discrepancy between the two figures obtained for the oxidation of the distillation residue from the first sample (53.5 and 58.5).

**Summary.**—Excepting in the case of the distillery and, inferentially, the brewery waste liquors, the results given in this paper require to be materially supplemented. Pending, however, detailed figures giving the rate of bacterial oxidation of the various kinds of trade waste, this oxidation being carried to its limit, the following wastes may in the meantime be roughly classified as follows:—

	Dissolved oxygen taken up from water in 5 days at 18° C. Parts per 100,000, by weight. About
"Standard" domestic sewage	33
Waste liquor from—	
Grain distilleries	33
Malt distilleries	33
Maltings and breweries	33
Calico printing and dyeing	25
Calico bleaching	(probably) 25
Paper mills	22
Tanneries	22
Jam factories	(P)10

In other words, unit section of bacterial filter will purify one volume of "standard" domestic sewage, or one volume of waste distillery liquor, or 25/33 volume of waste liquor from calico printing and dyeing, and so on, all the liquors being assumed as diluted to the same "strength" as the sewage.

Chemical Laboratory,  
30, High Street, Ealing, W. 5.

#### EXAMINATION OF SOME METHODS OF ASCERTAINING THE PURITY OF SACCHARIN.

BY PHYLLIS VIOLET MCKIE.

The saccharin prepared from toluene by the usual methods contains, before purification, small quantities of other sulphonic derivatives of toluene. The most common of these impurities are:—

- (1) *p*-Sulphamidobenzoic acid.
- (2) *Ortho*- and *para*-toluenesulphonamides.
- (3) *o*-Sulphamidobenzoic acid.—Statements in the literature regarding the isolation of this acid are misleading. It is described by Fahlberg (Ber., 1888, 21, 243) as being very easily soluble in water, and for that reason only isolated by ethereal extraction. 1



have, however, prepared this acid, and find that subsequent strong acidification of the mother liquor, which had been three times extracted with ether and gave by this means 81.5% of the theoretical yield of *o*-sulphamidobenzoic acid, deposited crystals of the same acid on standing representing a further 14%. It is therefore possible to "salt out" this acid with strong mineral acid. As saccharin is isolated by a similar "salting out," it is probable that the *o*-sulphamidobenzoic acid will, if present, be precipitated, to a certain extent with the saccharin.

(4) *Toluene-2,4-disulphonamide*.

(5) *Toluene-2,4-disulphonic acid*.

The presence of any of the above substances in commercial saccharin is variously indicated. Three criteria are of value in estimating the purity of saccharin:—

(a) *Depression of the melting point*.—The presence in saccharin of small amounts of impurity causes in several cases considerable depression (10°–15°) of the melting point.

(b) *Incomplete solubility in sodium bicarbonate*.—The presence of certain impurities is also indicated by incomplete solubility in aqueous sodium bicarbonate. Ortho and para-toluenesulphonamide and toluene-2,4-disulphouamide, in that they are extremely weak acids, are insoluble in this reagent. This property, moreover, as well as specifically indicating the presence of one or other of these substances, affords a means of eliminating them from the saccharin mixture.

(c) *Hydrolysis*.—Saccharin is hydrolysed in acid solution to *o*-sulphobenzoic acid and ammonia. Determination of the quantity of ammonia thus evolved from a known weight of commercial saccharin is, under certain conditions, a criterion of its purity.

(a) *Depression of melting point*.

The effect of each of the most common impurities, *p*-sulphamidobenzoic acid, *o*-toluenesulphonamide, and *o*-sulphamidobenzoic acid, on the melting point of saccharin has been examined.

The melting point of saccharin formerly given in the literature is 220° C. with partial decomposition (Mosso, Jahresb., 1887, 2585), but completely pure saccharin melts at 227°–227.5° C. (corr.) and shows no sign of decomposition; it re-solidifies and melts again at the same temperature. Richmond and Hill (J., 1919, 87) give m.p. 229° C.

*p*-Sulphamidobenzoic acid.—Mixtures of pure saccharin and *p*-sulphamidobenzoic acid of varying composition have been examined, and a diagram of the curve of the depression of the melting point of saccharin by successive additions of *p*-sulphamidobenzoic acid is given (cf. curve I.).

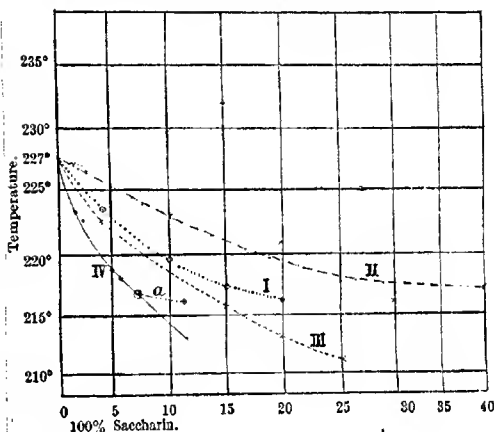
*Melting point of p-sulphamidobenzoic acid*.—Palmer (Amer. Chem. J., 4, 164) describes this acid as decomposing at 280° C. without melting, but this statement is erroneous. Even with markedly impure specimens such behaviour was never observed. Perfectly pure *p*-sulphamidobenzoic acid, prepared by repeated recrystallisation from water, has a sharp m.p. of 288°–289° C. (corr.), with no obvious sign of decomposition. On cooling, the substance partially re-solidified and melted again at 250°, showing that some decomposition had in actual fact occurred. This mixture again solidified, and a third melting point, showing marked decomposition, was observed at 300°.

It will be seen from the figure that the melting point is a fairly safe guide as to composition for mixtures containing from 0 to 10% of *p*-sulphamidobenzoic acid, the depression for which is normal, but the melting points of mixtures with a higher content of *p*-sulphamidobenzoic acid than 10% seem

to indicate abnormality, as is shown by the marked flattening of the curve in the region of 15% *p*-sulphamidobenzoic acid. Hence, in the case of mixtures of a lower melting point than 219.5° C. (the melting point of a 10% mixture) melting point is not a safe guide as to the content of *p*-sulphamidobenzoic acid.

*o*-Sulphamidobenzoic acid.—As before, the upper region of the curve of the depression of the melting point of saccharin by *o*-sulphamidobenzoic acid has been examined. The curve appears to be a straight line (curve II.), but in view of the fact that *o*-sulphamidobenzoic acid at a temperature of 180° changes into saccharin and *o*-benzoic ammonium sulphonate (Fahlberg, *loc. cit.*), the melting points of mixtures of this substance and saccharin cannot be taken as trustworthy evidence of the composition.

*o*-Sulphamidobenzoic acid + saccharin ————  
*p*-Sulphamidobenzoic acid + saccharin .....  
*o*-Toluenesulphonamide + saccharin ————  
Molar curve of Saccharin + *o*-toluenesulphonamide ————  
Molar curve of Saccharin + *p*-sulphamidobenzoic acid ————



Melting points of mixtures of saccharin with allied substances.

<i>p</i> -Sulphamidobenzoic acid.			<i>o</i> -Toluenesulphonamide.			<i>o</i> -Sulphamidobenzoic acid.		
%	Mols.*	M.p.	%	Mols.*	m.p.	%	m.p.	
10	0.04975	219.5°	5	0.02907	221.5°	10	223.0°	
15	0.07463	217.5°	10	0.05815	218.5°	25	221.0°	
20	0.09949	216.0°	20	0.11620	213.5°	35	216.5°	
						40	214.0°	

\* On the graph these numbers are shown multiplied by 100.

*o*-Toluenesulphonamide.—The depression of the melting point of saccharin produced by the presence of *o*-toluenesulphonamide is uniformly normal. Hence the melting point curve (curve III.) of these two substances is parallel to that of *p*-sulphamidobenzoic acid and saccharin up to the point at which the latter becomes abnormal, and since the molecular weights of *p*-sulphamidobenzoic acid (201) and *o*-toluenesulphonamide (171) are not appreciably different, is only separated from it by a difference of melting point of 1°–1.5° C.

The divergence of these two curves produced by the abnormality indicated in the melting point curve of *p*-sulphamidobenzoic acid and saccharin is clearly shown by consideration of the "equimolar" curves for these two pairs of substances (curves

IV.). The weight of each substance taken, divided by their molecular weight, gives the number of mols. which produce a depression corresponding to the observed melting point. Since equimolecular quantities of different substances produce equal lowering of melting point, the two curves should be congruent. But, as will be seen from the figure (IVa) that representing the melting point curve of *p*-sulphamidobenzoic acid and saccharin diverges sharply when 15% of *p*-sulphamidobenzoic acid is reached.

(b) *Solubility in sodium bicarbonate.*

Dilute, 6–8% sodium bicarbonate is added, drop by drop, to the powdered saccharin mixture until effervescence ceases, and solid ceases to be dissolved. After standing for some hours the mixture is filtered. Any toluenesulphonamide initially present in the crude saccharin is thus eliminated in this residual solid. The bicarbonate liquor contains the saccharin, plus any *o*- or *p*-sulphamidobenzoic acid present. Addition of moderately strong acid causes all these substances to be precipitated together. Saccharin comes down more slowly than does *p*-sulphamidobenzoic acid, but no separation by this means is practical.

On the other hand, when the bulk of the impurities has been removed by one of the standard procedures, this method can be conveniently used to obtain completely pure saccharin. The purified saccharin, m.p. not lower than 224° C., is dissolved in an equivalent quantity of aqueous sodium bicarbonate. The solution, filtered if necessary, is diluted with boiling water to twenty parts by volume for every one part of saccharin. The mixture, kept at a temperature of about 80° C., is very slowly precipitated with aqueous 5% hydrochloric acid in 30–50% excess. The addition of hydrochloric acid equivalent to 10 g. of saccharin took, under these conditions, six hours. In this way, and by slow cooling in a hot bath, large lustrous crystals with a sharp melting point of 227°–227.5° C. can be obtained.

(c) *Hydrolysis.*

The hydrolysis of saccharin to *o*-sulphobenzoic acid and ammonia by acids of certain strengths is well known, and a method for the estimation of saccharin is based on this reaction (Emmet Reid, Amer. Chem. J., 1899, 21, 461; Proctor, Chem. Soc. Trans., 1905, 87, 242). The method consists in boiling a known quantity of saccharin (0.3–0.5 g.) with *N*/1 hydrochloric acid (50 c.c.) for 2½ hrs. The ammonia is then estimated by the usual method. Experimental examination of this method showed an error of (1) –0.5%; (2) +0.2%; (3) –0.05%; (4) +0.3%, in four different estimations.

Of the sulphonic derivatives of toluene described above as common impurities of crude saccharin, all contain the group –NH<sub>2</sub>, and are therefore potentially capable of yielding ammonia on hydrolysis. That the validity of this method of estimation of saccharin might be fully tested, it was essential to ascertain whether the conditions favourable to

the hydrolysis of saccharin led also to the elimination of the –NH<sub>2</sub> group from these substances.

Proctor (*ibid.*) definitely states that the reliability of the process is not affected by the presence of any of the substances which are likely to occur as impurities in unpurified saccharin. This conclusion he bases on an examination of only *p*-sulphamidobenzoic acid, and *o*-toluenesulphonamide, which two substances he shows remain entirely unchanged under the conditions of the estimation. He did not, however, examine the behaviour of the three substances *p*-toluenesulphonamide, *o*-sulphamidobenzoic acid, and toluene-2,4-disulphonamide, all of which are possible impurities of saccharin. Proctor's results for *p*-sulphamidobenzoic acid and *o*-toluenesulphonamide have been confirmed.

*p*-Toluenesulphonamide.—Treatment of the pure amide with *N*/1 hydrochloric acid for 2½ hrs. led to no hydrolysis. In one case the acid liquor was made alkaline and distilled as in the usual Kjeldahl process; no ammonia was detected. In a second experiment the acid liquor after boiling was allowed to crystallise, when a solid with a melting point corresponding to that of pure *p*-toluenesulphonamide was recovered with no appreciable loss of weight.

*Toluene-2,4-disulphonamide.*—Again no hydrolysis was detected under the standard conditions. The amide does not crystallise out from the mother liquor on cooling, but extraction with ether gives a quantitative recovery of pure toluene-2,4-disulphonamide.

*o*-Sulphamidobenzoic acid.—This acid stands alone, in that it is hydrolysed to a very large extent—80%—under the standard conditions.

Of these substances, the most common impurity in unpurified saccharin is *p*-sulphamidobenzoic acid. This, as shown above, is not attacked by *N*/1 hydrochloric acid, and hence the recognised method of estimating saccharin holds, in general, fairly accurately. The presence of *o*- and *p*-toluenesulphonamide, and of toluene-2,4-disulphonamide also in no way affects the validity of the process. But the method is not capable of universal application since in the presence of *o*-sulphaminobenzoic acid (a not very uncommon impurity of saccharin) considerable inaccuracy may be introduced. Moreover, the specific detection of this acid is not easy.

In a modification of Proctor's method for the estimation of saccharin (Richmond and Hill, J., 1918, 246  $\tau$ ) the presence of this acid also invalidates the estimation. But in a later paper (*ibid.*, 1919, 8  $\tau$ ) the authors suggest a means of detecting and estimating this acid in mixtures with saccharin.

The above investigation was undertaken at the suggestion of Professor Orton, to whom I am indebted for direction and criticism.

University College of N. Wales.  
Bangor.











